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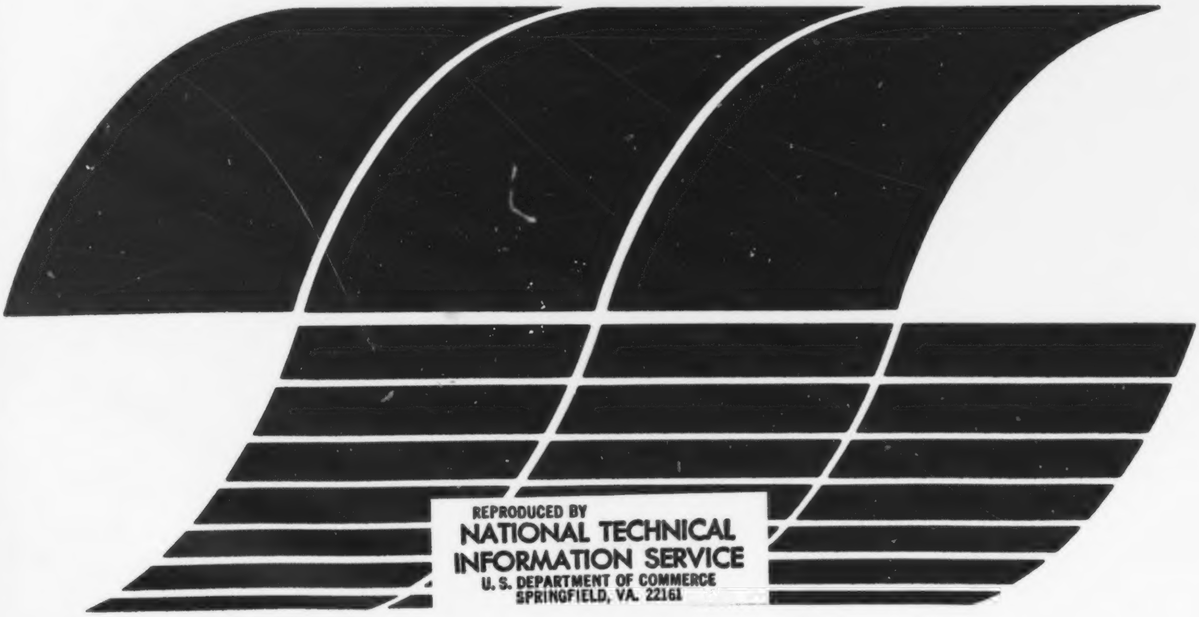

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16. ABSTRACT

The report describes a series of procedures for sizing dry aerosols and measuring H2SO4 entering and leaving the Shawnee flue gas desulfurization (FGD) prototype units. A Brink impactor was used to size dry particulate matter entering the FGD process. A manual system for the FGD process effluent was chosen on the basis of a literature survey, contacts with experts in the field, and an evaluation of available information. Chosen for the inlet was an FGD Meteorology Research Inc. cascade impactor. Finally, a method for H2SO4 vapor was developed which is based on the controlled condensation (Goksoyr/Ross) method. In addition to these procedures, a QA program was designed to ensure the overall quality of the data taken in the above procedures.

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Size Determination	Impactors	Particulate	14B	13I
Sulfuric Acid	Condensing	Brink Impactor	07B	
Sulfur Trioxide	Quality Assurance	Goksoyr/Ross Method		13H,14D
Vapors				
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FOREWORD

This manual has been prepared for the Industrial and Environmental Research Laboratory of the Environmental Protection Agency, Research Triangle Park, North Carolina, as part of Task 2 of Contract No. 68-02-2165.

The technical objective of this project was to prepare a series of procedure documents for sizing dry aerosols and measuring SO_3 entering and leaving a Flue Gas Desulfurization (FGD) unit and written for GS-4 personnel or equivalent. The sizing method for the dry particulate matter entering the FGD process will be a manual technique utilizing a Brink Impactor. A manual system for the FGD process effluent was chosen on the basis of a literature survey, contacts with experts in the field, and an evaluation of available information. The method chosen was the Meterology Research Inc. Cascade Impactor used out of stack. Finally a method for SO_3 (H_2SO_4 vapor) was developed based on the Controlled Condensation (Goksoyr/Ross) method and was successfully tested under laboratory conditions.

The project was divided into three areas of effort:

1. Aerodynamic Size Distribution Measurement of Dry Aerosols
2. Procedure for Sampling and Analysis of SO_3
3. Quality Assurance

Aerodynamic Size Distribution Measurement of Dry Aerosols

Documents were prepared describing the methods for determination of the size distribution of dry particulate matter at the inlet and outlet of flue gas desulfurization (FGD) process. The FGD process inlet measurement system was a Brink Impactor while the outlet measurement system, which must be suitable for extremely low grain loading, was selected from several candidate systems.

The selection of the outlet impactor system was based on the following criteria:

- Ease of assembly and operation - GS-4 level technicians should be able to operate the instrument.

- Ease of sample recovery - Sample removal should be accomplished under field conditions with minimum of effort.
- Construction material compatibility with sample and sampling environment - The equipment should not corrode or in any way contaminate the sample.
- Sampling period required for sample collection - Flow rates should be maximized to collect adequate amounts of sample for measurement in a reasonable sampling period under low grain loading conditions.
- Sample capacity - The system should be flexible enough to accurately size and collect particulate under high and low grain loadings.
- System design to minimize wall losses and re-entrainment - All samples should be deposited in collection trays or cups.

Applying these criteria, the MRI Impactor was selected. Procedure documents describing the operation of the Brink and MRI impactors are found in Chapters 1 and 2 respectively. These documents include: equipment lists, equipment assembly and preparation, on-site set-up and operation, sample removal and handling procedures, and sample weighing procedures. Other than making reference to known procedures (such as EPA Methods 1 through 4), this document will be designed to stand by itself and be directed toward GS-4 or equivalent personnel.

Procedure for Sampling and Analysis of SO_3

A procedure to sample and analyze for SO_3 in flue gas prior to and after FGD process was written. From TRW's knowledge of the SO_3 sampling problem, the Controlled Condensation (Goksoyr/Ross Coil), Brink Impactor and selective liquid impingement appeared to be the methods available. A literature evaluation of the systems was based on the following criteria:

- Sensitivity
- Selectivity
- Precision
- Accuracy
- Efficiency
- Ease of Operation
- Reliability/Maintainability
- Sample Recovery for Analysis

As a result of this evaluation the Controlled Condensation system was tested in the laboratory simulating the conditions in the FGD unit, and was found to be precise and accurate. Chapter 3 contains the document describing this procedure.

Quality Assurance

This effort was devoted to develop methods that will ensure the overall quality of the data taken in the above procedures. Chapter 4 describes general techniques associated with the dry aerosol sizing and SO_3 procedures as well as specific QA activities for each procedure. Included in the specific QA activities are:

- Critical checkpoint lists for each procedure
- Data validation procedures
- Maintenance schedules
- Troubleshooting procedures

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1. PROCEDURE FOR SAMPLING THE INLET OF AN FGD UNIT WITH A BRINK IMPACTOR

This method is for the determination of the aerodynamic size distribution of dry solids prior to the flue gas desulfurization (FGD) units at the TVA Shawnee Power Plant in Paducah, Kentucky. The Brink impactor with a specially designed internal cyclone is used to provide aerodynamic size distribution information between 0.3μm to 10μm in 6 distinct cuts. The recommended flowrate is 0.01 to 0.08 cfm.

This procedure uses the Brink impactor out of stack to sample the particulate from the gas stream entering the wet scrubber. After the large particles have been removed from the gas stream by the internal Brink cyclone, the remaining particles in the gas stream are then separated by a Brink Cascade impactor. By weighing each stage of the Brink impactor, the aerodynamic size distribution can be determined.

1.1 DOCUMENTS

1-1 Federal Register. 36(247):24888-9.

1-2 Brink BMS-11 Instruction Manual. Monsanto, Enviro-Chem Systems, Inc., St. Louis, Missouri.

1-3 McCain, J.D., A.N. Bird, and K.M. Cushing, "Field Measurements of Particle Size Distribution with Inertial Sizing Devices. Southern Research Institute, EPA 650/2-73-035, 1973.

1-4 Smith, W.B., K.M., Cushing, G.E. Lacey, and J.D. McCain. Particle Sizing Techniques for Control Device Evaluation. Southern Research Institute, EPA 650/2-74-102a, 1975.

1.2 EQUIPMENT AND MATERIALS

1.2.1 Sampling Equipment

- Brink impactor and 1 cfm pump (obtainable from Monsanto Enviro-Chem System Inc., St. Louis, Mo.)
- Aerotherm Isokinetic Flowrate Calculator (#HVSS-901, Aerotherm Corp., Mountain View, Ca.)

- Assemble a 5-foot probe from the following materials (see 1.4.1):
  1) Appropriate size Brink nozzle
  2) One Swagelok 1/4-inch SS male elbow (SS-400-2-4)
  3) 5-foot x 1/4-inch 304 SS tubing
  4) 4-1/2-foot x 1-1/2-inch OD x 0.035 inch wall aluminum tubing
  5) Two silicone rubber No. 8 stoppers (A. H. Thomas 8747-E83)
  6) Glass tape (Scotch glassfiber electrical heating tape)
  7) 50 feet of heater wire (S. Moore Co., Aurora, Ohio, #1659-40110)
  8) Two Omega (Stanford, Conn.) shielded thermocouples (I/C) (#TJ36-ICSS-18G-12 with a 12-foot lead)
  9) Two Omega (Stanford, Conn.) unshielded thermocouples (I/C) (#IRCO-032 with a 6-foot lead)
  10) Five Omega male connectors (ST-IRCO-M)
  11) One 6-foot heavy duty (~ 20A) electrical cord with a male plug
  12) Two 1-1/2 inch hose clamps
  13) 1/2-inch Teflon pipe tape
  14) Two square yards of asbestos cloth (VWR, Atlanta, Georgia, #10930-009)
- Stopwatch
- Heating mantle for impactor and filter (Glass-Col, Terre Haute, Ind.)
- Two wash bottles, one with distilled H_2O and one with acetone
- A source of 110V electrical power must be provided at the sampling location
- Five-place analytical balance
- Gelman, 47 mm inline filter housing (Product No. 2200, Gelman Inst. Co., Ann Arbor, Michigan)
- Reeve Angle 934-AH, 47 mm glassfiber filter (Reeve Angel Co.)

- One Dwyer Series 2000 magnehelic differential pressure gauge with the low temperature option for 0.30 in. H_2O (Dwyer Instr. Inc., Michigan City, Ind.).

- One Dwyer Series 2000 magnehelic differential pressure gauge with the low temperature option for 0-60 in. H_2O.

1.2.2 Coating and Weighing Materials

- Apiezon H grease.
- Drierite, 5 lb.
- Large desiccator, Kimble #21050, 250 mm min. diameter with porcelain plate.
- Petri dishes (top and bottom), 60 x 15 mm pyrex.
- Camel hair brush.
- Tweezers.
- PVC gloves, U.S. Industrial Gloves, Compton, California.
- Whatman No. 1 paper sheets (46 x 47 cm).
- Kimwipes.
- Acetone, reagent grade.
- 3-inch rubber plug with a 1.5 inch hole drilled in the center.
- Toluene, reagent grade.

1.3 REQUIREMENTS

1.3.1 System Design

The Brink sampling train consists of a 5-ft. x 1/4-in. ID 304 stainless steel probe, Brink internal cyclone (based on SoRI design), five impactor stages, a Gelman 47 mm filter holder and a 934-AH Reeve Angel filter, three impingers, a pump and a calibrated orifice (see Figure 1).

1.3.2 Sampling Procedure

The flow rate through the impactor determines the size cut-off that each stage will collect. As will be described in Section 1.4, an average isokinetic sampling rate will be determined. Once the average flow rate

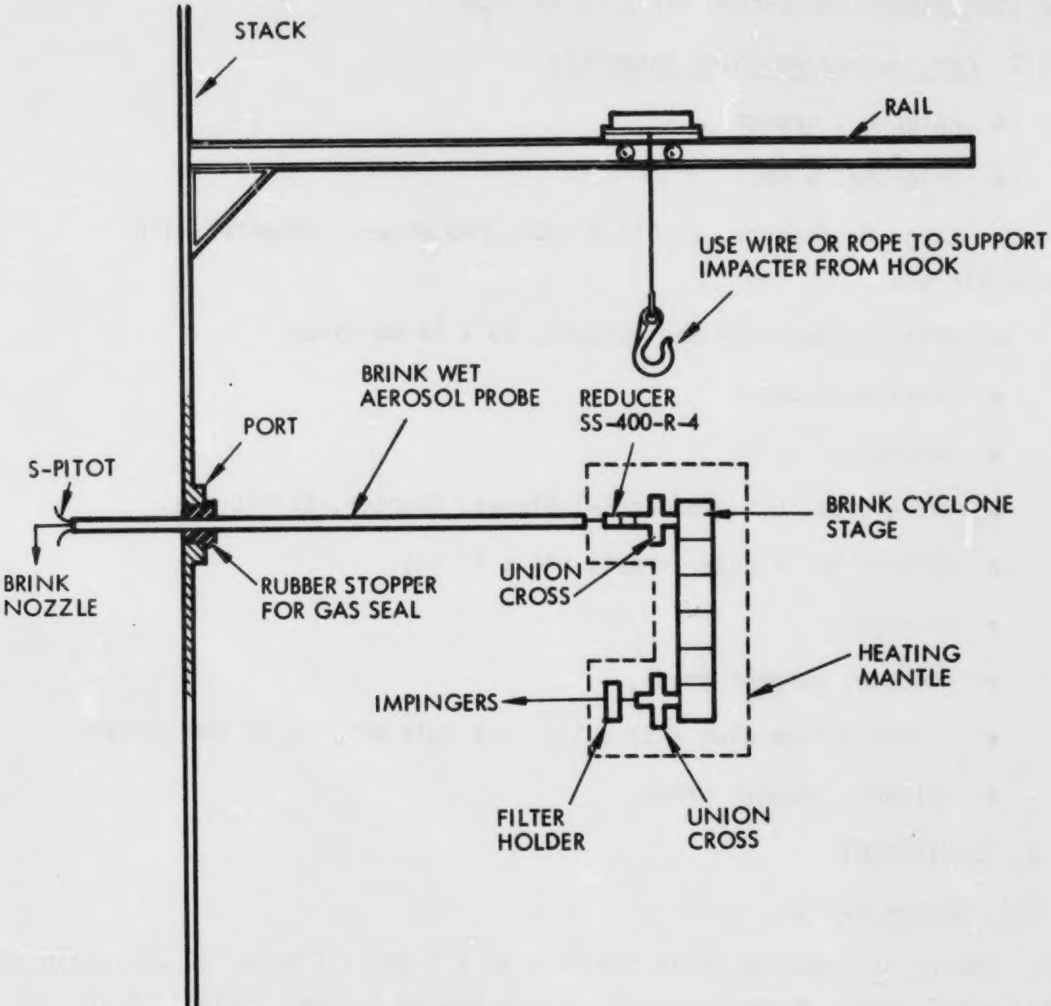

STACK
RAIL
USE WIRE OR ROPE TO SUPPORT IMPACTER FROM HOOK
BRINK WET AEROSOL PROBE
REDUCER SS-400-R-4
PORT
S-PITOT
BRINK CYCLONE STAGE
BRINK NOZZLE
RUBBER STOPPER FOR GAS SEAL
UNION CROSS
HEATING MANTLE
IMPINGERS
FILTER HOLDER
UNION CROSS

Figure 1. Brink Impactor

is established, it must be maintained throughout the run regardless of the individual velocities at each point in the traverse.

1.3.3 Handling

Care must be taken to limit contact with the stages. At no time after cleaning should the stages be touched with ungloved hands. All of the laboratory manipulations are to be done in a clean environment using tweezers to handle the stages. Remember, several grains of dust could represent the total weight captured on the stages. Contamination control is essential during greasing, drying, and weighing.

1.3.4 Calibration and Maintenance

After each run, the probe nozzle, probe, connecting lines, S-pitot tubes, impactor, and impinger system must be cleaned. After the run, the probe and connecting lines are rinsed with reagent grade acetone. The S-pitot tube should be backflushed with a high pressure air line. The impactor cleaning procedures are detailed in 1.4.4. The impinger system is flushed out and the proper solvents replaced in the impinger bottles prior to the next run.

Besides these daily procedures, the S-pitot C_p and the ΔH@ of the flowmeter orifice are determined every 3 months. If any evidence of corrosion appears (pitting, scale build-up, etc.), the C_p and ΔH@ recalibration procedure should be repeated as needed or the part returned to the manufacturer. The Brink impactor must have an up-to-date ΔP vs cfm calibration chart. This chart is supplied by the manufacturer or can be determined experimentally. See Tables 7 and 8 for further information on equipment care and maintenance.

1.3.5 Cleanliness

Gas carrying lines should be cleaned weekly. In particular, no particulate build-up in the pitot tube can be tolerated. The probe, lines, and impactor must be completely clean before use.

1.3.6 Safety

OSHA safety requirements as regards to working environment and operator safety will be met at all times. The reagents mentioned in the procedure are not extremely toxic, but misuse of any chemicals can be harmful.

1.4 PROCEDURE

1.4.1 Probe Manufacture

Refer to Figure 2. The necessary equipment is listed in paragraph 1.2.1. The following instructions are used for the construction of a 5-foot probe. At all times follow correct electrical safety procedures. Be sure that no sharp pieces of metal abrade any of the electrical wires.

a) Cut the 304 SS 1/4-inch tubing to 4.5 ft.

b) Attach the male elbow to one end of the probe.

c) The proper Brink nozzle will be screwed into the male thread of the elbow prior to sampling.

d) Align the shielded thermocouple as shown in Figure 2. Using the glass tape, secure the shielded thermocouple to the probe. Approximately halfway down the probe from the Swagelok elbow, attach the unshielded thermocouple. Continue down the probe, securing both thermocouple leads simultaneously against the tube.

NOTE

Be careful never to kink
thermocouple or thermocouple
leads.

e) Approximately three inches from the end of the tube, place a final wrapping of glass tape.

f) Take 12 feet of heating wire and fold it in half.

g) Beginning six inches from Swagelok union, wrap the probe with the doubled up heating wire. Make sure the heating wire is snug up next to the probe and secured every six inches with a wrapping of glass tape. Do not lay the coil of the heating wire on the tip of the unshielded TC. Simply gauge the wrapping to place the TC in one of the gaps between coils. Secure the heating coils to either side of the TC with tape to prevent them from slipping over the TC. Wrap the coils close enough so that the heating wire is completely used up three inches from the end of the probe. Secure the end of the heating tape with a final wrap. Wrap one layer of asbestos cloth around the heating tape.

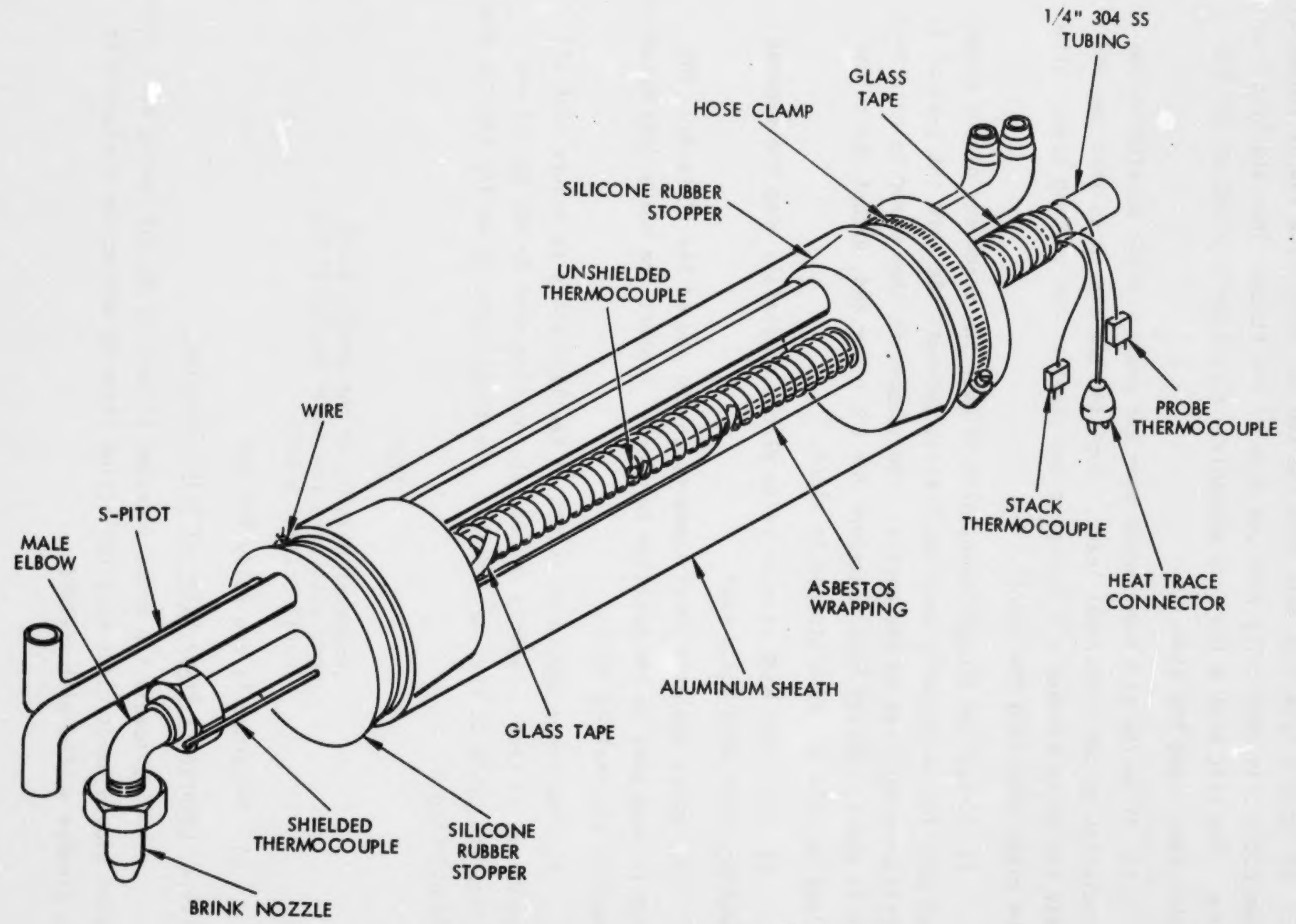

1/4" 304 SS TUBING
GLASS TAPE
HOSE CLAMP
SILICONE RUBBER STOPPER
UNSHIELDED THERMOCOUPLE
WIRE
PROBE THERMOCOUPLE
STACK THERMOCOUPLE
HEAT TRACE CONNECTOR
S-PITOT
MALE ELBOW
ASBESTOS WRAPPING
ALUMINUM SHEATH
GLASS TAPE
SHIELDED THERMOCOUPLE
SILICONE RUBBER STOPPER
BRINK NOZZLE

Figure 2. Schematic of Brink Sampling Probe

h) Bore a 5/16-inch hole into the two No. 8 silicone rubber stoppers, then cut a slit vertically down one side of the stopper into the 5/16-inch hole. The slit will allow easy assembly. Also provide a cutout for the pitot tube along the side.

i) Slide the aluminum sheath over the probe. Avoid scratching the insulation on the electrical leads. Position the sheath so that the end near the elbow extends ∿ 1 inch past the start of the heating tape. Slide the pitot tube into the sheath.

j) Spread the stopper open, slip it over the stack end of the probe, and position it properly over the S-pitot cut-out. Be sure the S-pitot is positioned parallel to the nozzle. The stopper is then wired to help hold it in place. Repeat this procedure for the other end, except use a hose clamp to hold the back stopper in place.

k) After the back stopper is in place, completely wrap the exposed heating coils with glass tape.

l) Place the male quick connects on the end of the TC leads. The red TC lead goes to the negative terminal. Connect the heavy duty extension cord to the heating tape.

m) The probe should be tested in the laboratory to ensure that all parts are in order. Simply connect the heating wire to the Variac and allow the probe to heat up. Monitor the temperature to verify the TCs are functioning.

NOTE

> Whenever heating up the probe, start off with very low power inputs (∿ 5%) until heating starts.

n) The probe is now ready for use.

1.4.2 Laboratory Preparation of Brink Impactor

a) Disassemble the Brink impactor (Figure 3) by unscrewing each stage. With gloved hands clean each collection plate by wiping the surface with a Kimwipe wetted with acetone.

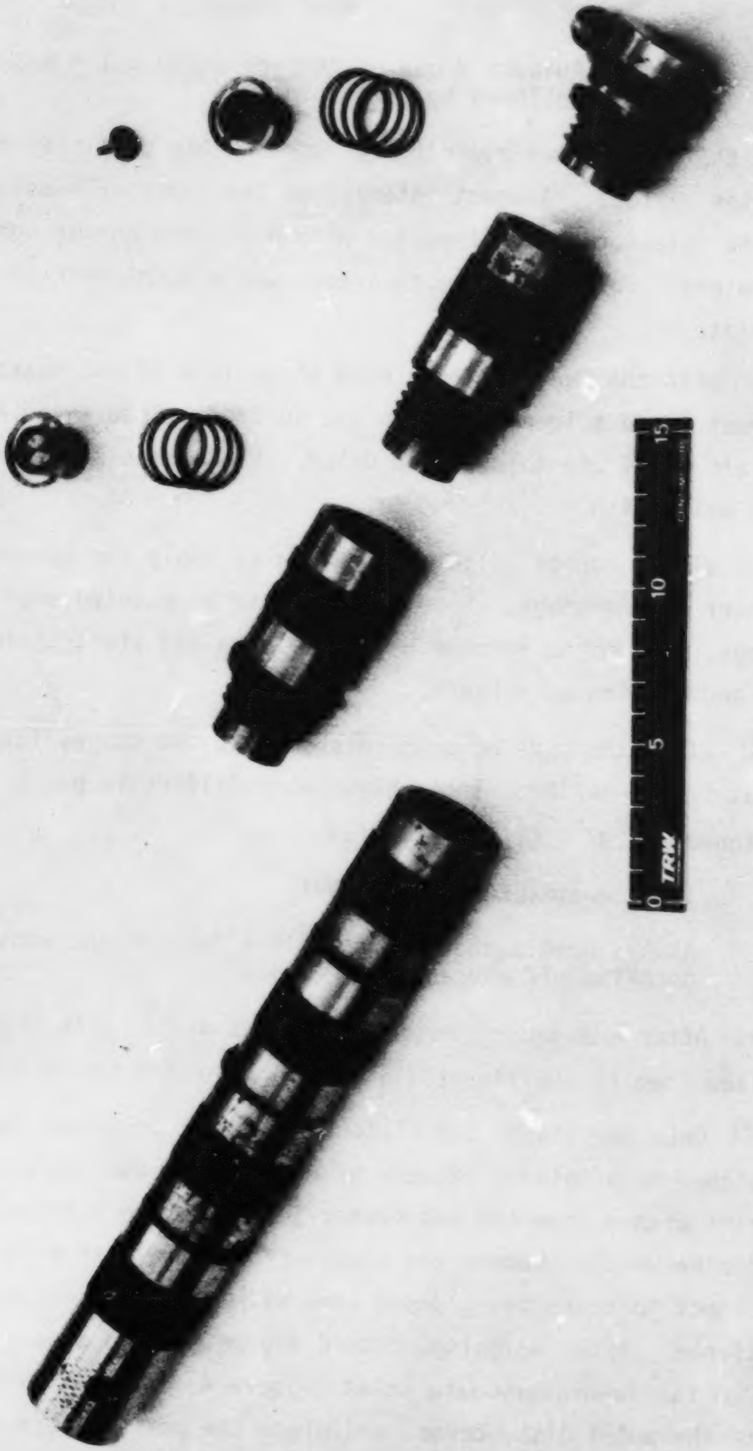


Figure 3. Brink 5-Stage Impactor Disassembled

NOTE

> Residual Apiezon H can be removed using a 1 m NaOH solution followed by toluene.

Inspect the plate after cleaning for particulate or finger marks on the collection surface. Inspect interior of the impactor housing for particulate. Clean the interior of the impactor with a squeeze bottle containing acetone, and Kimwipes. For hard-to-reach areas, use a camel-hair brush to remove the particulate.

b) With the tweezers, dip each stage in a 100 ml beaker with ~50 ml of reagent grade toluene to clean the surface. Remove and hold the stage in the air until the toluene has dried. Place each stage in a separate labeled petri dish.

c) With a rubber policeman, carefully apply the Apiezon H grease to the center of the stage. Should the grease be painted over the edge of the stage, remove the Apiezon H with toluene and start again. Coat six stages and use one as a blank.

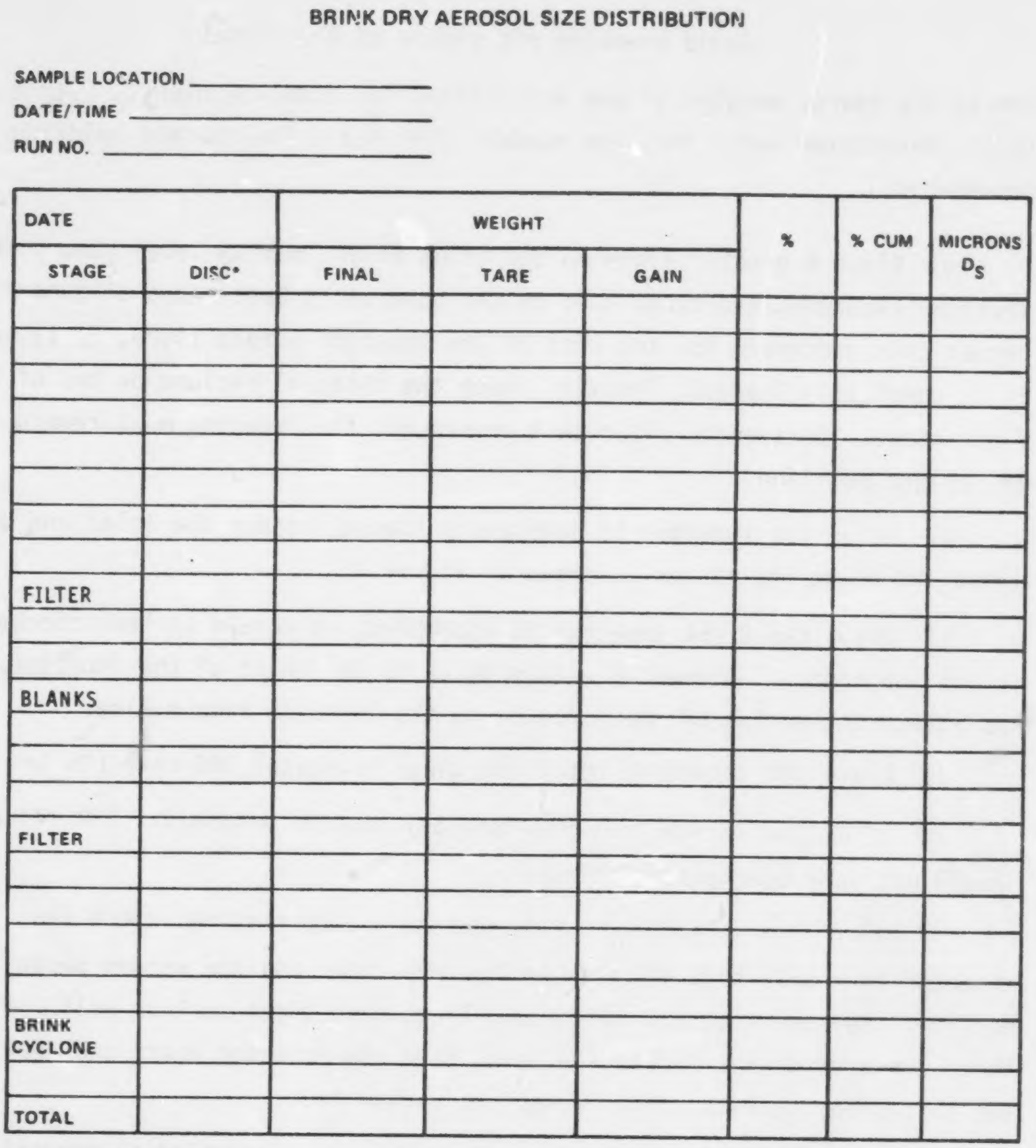
d) Place the covered petri dishes with the stages into an oven for four hours at 175°C (347°F). Three glassfiber filters in petri dishes are conditioned at 287°C (574°F) for four hours.

NOTE

> Always handle the filters with a tweezer and avoid breaking off pieces of the filter.

e) After four hours, remove the petri dishes with stages and filters and allow them to equilibrate in a desiccator for two hours.

f) Once the stages and filters have been dried and desiccated, they are weighed on a balance capable of weighing to the nearest 0.01 mg. Remove the petri dishes from the desiccator just prior to weighing (keep desiccator closed otherwise). Remove the stages from petri dish with a tweezer being careful not to touch the greased area with the tweezers, and place them on the balance. After weighing, record the weight and disc number (on petri dish) on the laboratory data sheet (Figure 4) and place the coated disc back in the petri dish, cover, and place the petri dishes near the impactor in a clean, dust-free area. Weigh a 47 mm filter to the nearest 0.01 mg and immediately load it into the filter holder.



BRINK DRY AEROSOL SIZE DISTRIBUTION

SAMPLE LOCATION ____________________
DATE/TIME ____________________
RUN NO. ____________________

DATE		WEIGHT			%	% CUM	MICRONS D_S
STAGE	DISC*	FINAL	TARE	GAIN			
FILTER							
BLANKS							
FILTER							
BRINK CYCLONE							
TOTAL							

% - Weight gain on each stage divided by the total weight gain.

CUM% - Starting with the filter accumulate each stage to arrive at the cumulative percent smaller than the previous D_S.

* - Disc Code for labeling petri dishes should be the date of run, stage no. and run letter series (example: 8/27/75, 1A; 8/27/75, 2A; etc.). The letters series represents the sequential number for each successive run that day: 8/27/75, 1A; 8/27/75, 1B would be the next run.

** - As corrected by equation (3).

Figure 4. Brink Laboratory Data Sheet

NOTE

Avoid breaking off pieces of the filter.

Retain the spare, weighed stages and filters for handling blanks. Store them in the desiccator until they are needed. (See 4.1.8 for correct weighing procedures.)

g) Place a greased stage on the Brink #5 collection level (the bottom section) recording the stage used on the Laboratory Data Sheet (Figure 4). Repeat this procedure for the rest of the impactor stages (4, 3, 2, 1) until it is completely loaded. Finally, place the internal cyclone on top of the first stage. During and after this procedure, the impactor must remain in an upright position.

h) After the impactor is completely loaded, attach the inlet and outlet lines including the filter as shown in Figure 5.

i) After the Brink impactor is assembled, it should be leak checked in the laboratory. Connect a vacuum gauge to the inlet of the impactor, and attach the outlet of the impactor to the in-house vacuum line.

j) Leave the vacuum on until the gauge indicates 380 torr (15 in. Hg).

k) Close the vacuum line and note any rise in pressure. The vacuum should not vary over several minutes.

l) If a leak is noted by a decreasing vacuum reading, check the impactor to verify that all connections are tight and the vacuum gauge is working. Be sure that all the vacuum lines have tight seal as well. If these measures do not locate the leak, take the impactor apart and replace any suspicious gaskets, then repeat the vacuum test.

Once the impactor is leak checked, both ends are sealed to prevent dust from entering, and the impactor is placed inside of the heating mantle and taken to the sampling site.

1.4.3 Measurements and Calculations for Isokinetic Sampling

a) The duct geometry must be first considered. For the circular 40-inch diameter ducts at the Shawnee limestone wet scrubber, refer to Figure 6.

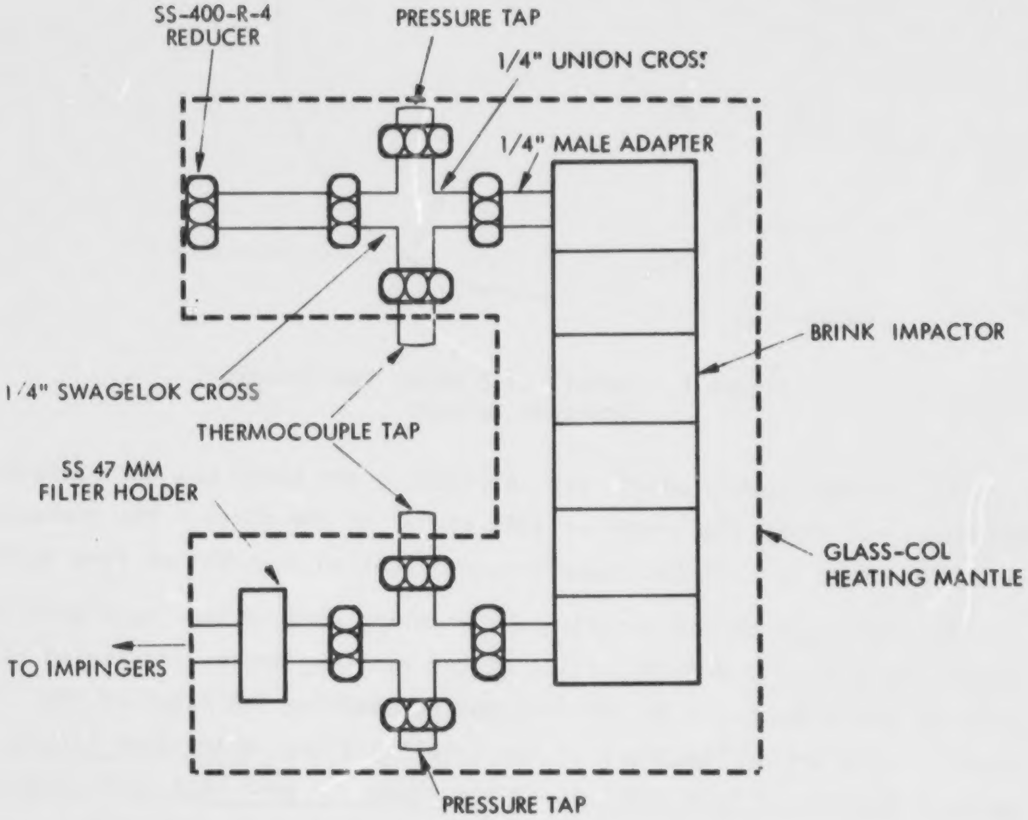

SS-400-R-4
REDUCER
PRESSURE TAP
1/4" UNION CROSS
1/4" MALE ADAPTER
BRINK IMPACTOR
1/4" SWAGELOK CROSS
THERMOCOUPLE TAP
SS 47 MM
FILTER HOLDER
GLASS-COL
HEATING MANTLE
TO IMPINGERS
PRESSURE TAP

Figure 5. Brink Impactor and Filter Assembly

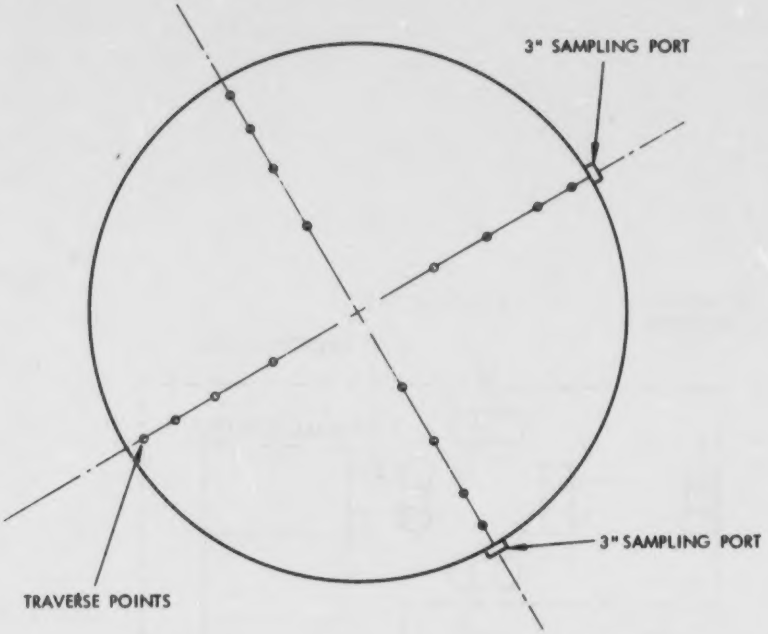


Figure 6. Detail of 8 Point Two Diameter Traverse Pattern

b) Sixteen sample points are selected; eight along one axis across the duct, and eight along another axis at 90° to the first. The distance along the probe to mark each sample point location is obtained from Table 1.

c) Test site should consist of a sampling port in the stack with an opening to allow the easy insertion of the sampling probe, and sealed to minimize the disturbance of the flow during sampling. Because of the negative pressure in the stack at the sampling sites, extra care should be taken in ensuring a good seal around the probe. A poor seal will lead to low temperatures at the first sampling point. The electrical power required to operate the equipment must be available is approximately 35 amp/115V.

1.4.4 Isokinetic Operation of the Brink Impactor

a) Prior to initiation of sizing experiments, the S-pitot probe is to be recalibrated. The C_p is noted and entered into the Field Data Sheet (FDS), Figure 7.

1. ΔP AND T_S TRAVERSE DATA

SAMPLE PORT	TRAVERSE POINT	ΔP IN. H_2O	$\sqrt{\Delta P}$ IN. H_2O	T_S, °F
$(\sqrt{\Delta P})^2$ =		AVERAGE	$\overline{\sqrt{\Delta P}}$ =	$\overline{T}_S$ =

2. BRINK OPERATIONAL VARIABLES

VARIABLE	VALUE
C_p	
P_S	
$(\overline{\sqrt{\Delta P}})^2$	
$\overline{T}_S$	
% H_2O	
D_N	
MW_2	29.5
V_S	
ΔP_b	
ΔP_c	

Figure 7. Brink Field Data Sheet

-Continued-

3. **FIELD DATA SHEET**

PLANT ____________________

DATE/TIME ____________________

SAMPLING LOCATION ____________________

RUN NUMBER ____________________

OPERATOR ____________________

AMBIENT TEMPERATURE ____________________

BAROMETRIC PRESSURE ____________________

STACK PRESSURE, (P_S) ____________________

FILTER NUMBER ____________________

INLET GAS FLOW ____________________

PROBE HEATER SETTING ____________________

SYSTEM LEAK RATE ____________________

MOISTURE

Impingers

Final vol. ____________ ml

Initial vol. ____________ ml

Net vol. ____________ ml

Silica Gel

Final wt. ____________ g

Initial wt. ____________ g

Net wt. ____________ g

Total H_2O

Impingers ____________ ml

Silica gel ____________ g

Total ____________ g

SCHEMATIC OF TRAVERSE POINT LAYOUT

READ AND RECORD ALL DATA EVERY____ MINUTES

TIME	STACK TEMPERATURE, (T_S), °F	PITOT TUBE ΔP, in. H_2O	PUMP VACUUM, in Hg.	IMPACTOR TEMP. °F		SKIN TEMPERATURE, °F	INLET STATIC PRESSURE P_{IM}, in. H_2O	PRESSURE DROP ACROSS BRINK CORR. TO STACK ΔP_C, in. Hg.
				IN	OUT			
AVERAGE								

Figure 7. Brink Field Data Sheet

TABLE 1. LOCATION OF TRAVERSE POINTS IN CIRCULAR STACKS (PERCENT OF STACK DIAMETER FROM INSIDE WALL TO TRAVERSE POINT)

Traverse Point Number on a Diameter	Number of Traverse Points on a Diameter		
	5	8	10
1	4.4	3.3	2.5
2	14.7	10.5	8.2
3	29.5	19.4	14.6
4	70.5	32.3	22.6
5	85.3	67.7	34.2
6	95.6	80.6	65.8
7		89.5	77.4
8		96.7	85.4
9			91.8
10			97.5

b) Using the S-pitot probe and referring to Figure 6 and Table 1, perform a velocity traverse at the sampling site. At each point, record the ΔP and stack temperature (T_S), and ΔP on the FDS. After the velocity traverse, be sure to clean the probe of any debris.

c) Determine the average temperature (T_S - ^oF) and the average $\overline{\Delta P}$ $\left[(\sqrt{\overline{\Delta P}})^2\right]$ in inches of H_2O. Record these values on the FDS.

d) Using the Isokinetic Flowrate Calculator complete Table 2 on the FDS. (Note that only the velocity (V_S) will be calculated. For detailed instructions on how to use the Isokinetic Flowrate Calculator, see Appendix A. The following are the condensed instructions to be found on the back of the calculator. Definitions of terms are listed in the paragraph below and also on the calculator.)

1) Reset Hairline over C_p.

2) Set $\overline{T}_S$ at Hairline and move Hairline over V1 arrow.

3) Set V2 arrow under Hairline and move Hairline over $(\sqrt{\overline{\Delta P}})^2$.

4) Turn over calculator without moving Cursor and set M_S under Hairline.

5) Read Stack Velocity V_S at stack pressure P_S.

6) Record all this data on Table 2 on the FDS (Figure 7).

If the flowrate calculation is off scale on the Aerotherm calculator, then use this formula:

$$V_S = 85.48\ (C_p)\ (\sqrt{\Delta P})\sqrt{\frac{T_S + 460}{P_S\ M_S}} \qquad (1)$$

e) From the nozzle selection chart (Figure 8) and V_S select a probe nozzle that will give a sampling rate (F) between 0.01 and 0.08 cfm. Use the largest nozzle (D_N) possible without exceeding 0.08 cfm. Record the D_N on Table 2 on the FDS.

NOTE

A 1.5 mm nozzle is the recommended size.

f) Using the calibration curve for the Brink Model B cascade impactor, select a ΔP_b corresponding to F (Figure 9).

g) Calculate the ΔP_c under stack conditions.

$$\Delta P_c = \Delta P_b \left(\frac{M_S}{29.0}\right)\left(\frac{537}{460 + T_S}\right)\left(\frac{P_S}{29.92}\right) \qquad (2)$$

h) Record these values on Table 2 on the FDS (Figure 7).

i) Symbols:

C_p = Pitot tube coefficient (in. H_2O).

$\overline{T}_S$ = Average stack temperature (^oF)

P_S = Stack pressure (in. Hg)

P_{IM} = Static pressure read at inlet to Impactor (normally in. H_2O)

P_{IA} = Static pressure read at inlet to Impactor converted to absolute pressure

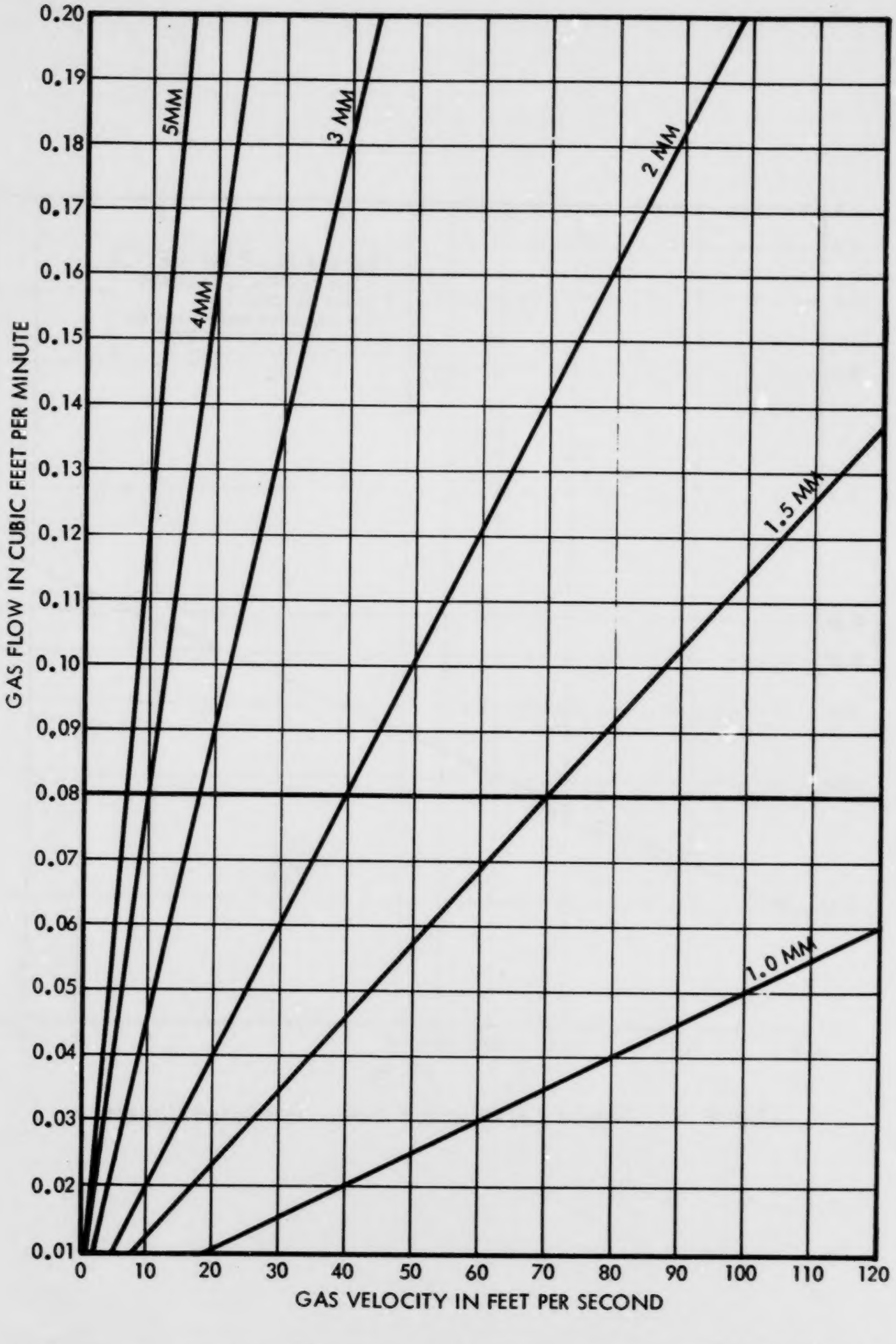


Figure 8. Gas Velocity Versus Gas Flow for Several Nozzle Sizes

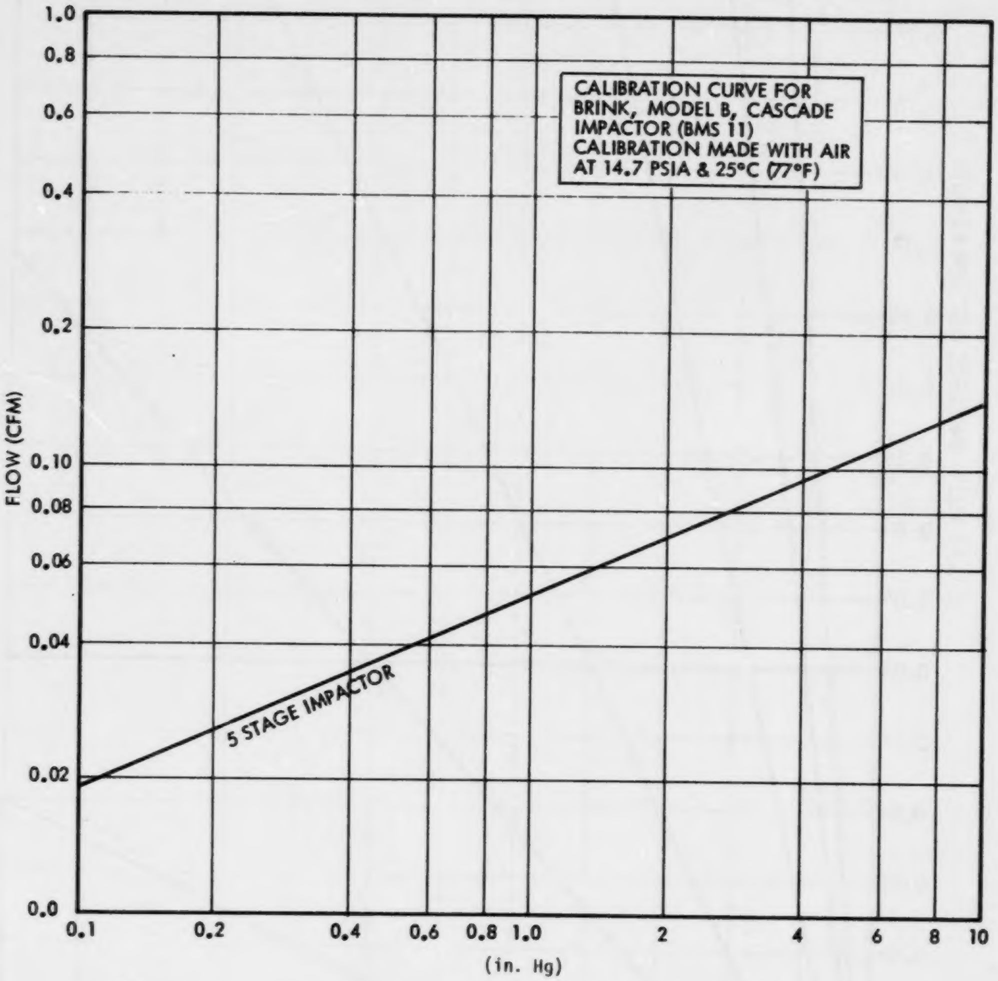


Figure 9. Sample Calibration Curve for Brink Impactor

D_N = Nozzle diameter (in.)

$\Delta\bar{P}$ = Average pitot ΔP (in. H_2O)

ΔP_b = Pressure drop across Brink Impactor at standard conditions, in. Hg

ΔP_c = Pressure drop across the Brink Impactor corrected to stack conditions, in. Hg

ΔP_a = Pressure drop across the Brink impactor during the run.

M_S = Molecular weight of the stack gas

V_S = Average velocity in the stack

F = Brink sampling rate (cfm)

NOTE

Capital subscript (S) refers to stack conditions.

1.4.5 Site Equipment Setup and Operation

a) In the 3-inch port insert a 3-inch plug with a 1.5-inch hole drilled into the center. Another 3-inch plug is used to prevent gases from escaping from the wet scrubber prior to the insertion of the probe. Clean the probe with a brush and rinse with acetone to ensure that all particulate matter is removed prior to the run.

b) Support the Brink system as shown in Figure 1.

c) Connect the 0-60 in. H_2O magnehelic gauge across the impactor. The 0.30 in. H_2O magnehelic gauge is connected to the inlet of the impactor. Be sure that the high and low pressure taps are connected to the correct inlets of the gauge.

d) Connect all the thermocouples into the readout.

e) Make sure that all of the connections that have been made are tight.

f) Put the correct nozzle on the end of the probe.

g) Connect Brink impactor to probe via reducer fitting. If a 1/4-inch reducer is not available, use a 1/4-inch union and a short (∿ 3-inch) piece of 1/4-inch tubing to go with the union to the union cross.

h) Connect the soap bubble flow meter to the vacuum pump exit. Be sure that the bubble flow meter is vertical. Close off the end of probe with a stopper and turn on the vacuum pump and adjust the vacuum to read 380 torr (15-inch Hg).

i) Begin measuring the flow rate with the bubble gauge. If the leak rate is less than 23 mL/min. (0.0008), then the system is ready for use. If a leak rate greater than 23 mL/min. is found, the system should be checked for loose joints and connections. The pump should also be checked and any worn parts replaced. (See Tables 7 and 8 for further information).

j) After the leak check, begin to heat the probe and impactor to the highest stack temperature plus 12°C (25°F), but not higher than 175°C (347°F). Do not overheat the impactor or weight loss problems will occur with the greased stages.

k) When probe and impactor are 12°C (25°F) above stack conditions, the run can start.

NOTE

Maximum use temperature for the probe is 400°F.

l) Turn the pump on and immediately insert probe into the stack until it is at the first sampling point. Be sure the S-pitot and nozzle are parallel to the gas flow. Immediately adjust the ΔP across impactor at ΔP_c. Record ΔP_c, T_S, P_{IM}, skin temperature, I/O gas temperatures from impactor, T_m, gas meter readings, and ΔP from the pitot.

Sample at each point for 45 seconds. Be sure to complete all other information on the FDS.

m) At the end of the run, the probe is removed from the stack and the pump is shut down. Slowly close the flow control valve to dissipate any back pressure in the system and thus prevent water from surging forward in the impinger system. Stop the stopwatch and record final gas meter reading on field data sheet.

n) Using gloves, remove the Brink Impactor with heating mantle and return it immediately to the library. Cover the inlets to the impactor to prevent particles from entering or leaving.

NOTE

> During this period, avoid jarring the impactor. Extreme care should be taken to avoid the addition or loss of collected particulate. Carry the impactor upright and do not expose it to dust.

o) Carefully rinse the probe with reagent grade acetone collecting the rinse until a clean stream of liquid issues from the probe.

NOTE

> Take extreme care in performing this task as the small amounts of particulate matter recovered represent a large portion of the total particulate aerosol collected. Both contamination and loss of sample must be avoided. Any accidents which occur must be recorded on the field data sheet.

p) Once the impactor is transferred to laboratory, clean the outside of the impactor of any dust. This should be done in the prep room prior to entering the clean room.

q) Be sure that the correctly labeled petri dishes are nearby, ready to accept the collection plates as they are removed.

r) With the impactor in an upright position, begin to remove the housing starting at the top. Remove the cyclone cup and place it in the correct petri dish. Inspect the inlet nozzle for any sign, no matter how little, of particles collected on the walls. Note the presence of the particulate matter on the back of the laboratory data sheet.

s) If any particles are found, they should be carefully brushed onto the collection plate below their collection point.

t) Inspect all the nozzles for any sign of pitting or corrosion. Especially inspect the sides of the nozzle for particles that might have collected there. If any particles are found, note this fact along with a description of any patterns formed, color, or quantity obtained on the back of the laboratory data sheet.

u) Carefully brush these particles onto the collection plate below the nozzle.

v) Repeat these activities for all the stages and the filter. In the filter's case be sure that all fragments of the filter are removed from the filter support; even the loss of the smallest fragment can affect the weight of the filter.

w) Desiccate the collection plates and filter for 2 hours.

x) Rinse the connecting lines from the probe to the impactor with acetone until a clean stream is obtained. Add this rinse to the probe rinse.

y) Rinse the connecting lines from the impactor to the filter housing with acetone. The particulate weight after evaporation is added to the filter.

z) Evaporate enough of the acetone from the probe and line rinses so that all the particulate and the remaining acetone can be quantitatively transferred to a tared 30 mL beaker. At all times handle the 30 mL beaker with gloved hands.

aa) Evaporate the bulk of the acetone from the 30 mL beakers on a hot plate allowing the rest of the acetone to air dry in a clean, dust free area. Dry the particulate at $110^{o}C$ for two hours and desiccate with the rest of the samples for approximately 2 hours.

bb) Weigh the collection plates and filter to the nearest 0.1 mg. At the same time weigh the balance and sample blanks (see section 4.1.8 for specific procedures to correct for any weighing errors). Weigh the probe and line rinses. Record the data on the laboratory data sheet (Figure 4).

1.5 DATA REDUCTION

NOTE

A computer program for this data reduction is
available from Monsanto Enviro-Chem, St. Louis, Mo.

1) Table 2 contains the known data; enter the field data in Table 3.

NOTE

(Lower case (s) subscript refers to an impactor stage.)

TABLE 2. CALCULATION OF PARTICLE SIZE CUT-OFFS, KNOWN DATA

Known Variables	Data
Density of Aerosol Particle (g/cc)	$\rho_p \simeq 1$
Molecular Weight of Sample Gas	$MW_2 = 29.5$
Molecular Weight of Calibration Gas	$MW_1 = 29.0$
Temperature of Gas at Calibration Conditions, °K	$T_1 = 298$
Static Pressure Under Calibration Conditions, atm.	$P = 1.0$
Gas Viscosity at Sampling Conditions (323°F), poises	$\mu = 2.18 \times 10^{-4}$
Stage Jet Diameter, cm	$D_{c_1} = 0.249$
	$D_{c_2} = 0.1775$
	$D_{c_3} = 0.1396$
	$D_{c_4} = 0.0946$
	$D_{c_5} = 0.0731$
Dimension Conversion Constant	$g_c = 1$

TABLE 3. BRINK DRY AEROSOL-SIZE DISTRIBUTION (CALCULATION OF PARTICLE SIZE CUTOFFS, CALCULATED DATA)

Unknown Variables	Calculated Data
Pressure Drop Across the Impactor During Test (in. Hg)	ΔP_c =
Effective Pressure Drop (in. Hg)	ΔP_E =
Flowrate in Impactor During Sampling (cc/sec)	F =
Barometric Pressure (in. Hg)	P =
Pressure at Inlet to Brink Impactor (manometer reading)	P_{IM} =
Pressure at Inlet to Brink Impactor Corrected to Absolute (in Hg)	P_{IA} =
Pressure at Inlet to Impactor Corrected to Absolute (atm.)	P_{IA} =
Density of Gas at Inlet Sampling Conditions (g/cc)	ρ_I =
Average Temperature of Gas at Sampling Conditions (°K)	T_S =
Pressure (P_s) at Outlet of Each Stage (atm)	P_1 =
	P_2 =
	P_3 =
	P_4 =
	P_5 =
Density (ρ_S) of Gas Out of the Various Stages (g/cc)	$\rho 1$ =
	$\rho 2$ =
	$\rho 3$ =
	$\rho 4$ =
	$\rho 5$ =
Characteristic Diameter (D_s) (microns)	D_1 =

Continued

TABLE 3. BRINK DRY AEROSOL-SIZE DISTRIBUTION (CALCULATION OF PARTICLE SIZE CUTOFFS, CALCULATED DATA) (Continued)

Unknown Variable	Calculated Data
Characteristic Diameter (D_s) (microns) (Continued)	D_2 =
	D_3 =
	D_4 =
	D_5 =
Cumulative Percentages	Σ_6 =
	Σ_5 =
	Σ_4 =
	Σ_3 =
	Σ_2 =
	Σ_1 =

Test date/time_______________

Sample Location_______________

Run Number__________________

2) Using the average actual $\Delta\bar{P}_a$ maintained during the run, determine effective pressure drop, ΔP_E, using equation (1-1)

$$\Delta P_E = \Delta P_a \left[\frac{MW_1}{MW_2} \times \frac{\bar{T}_I}{T_1} \times \frac{(29.92)}{P_{IA}}\right] \qquad (1\text{-}3)$$

where $\bar{T}_I$, is the average impactor in/out gas temperature in ^oK.

$$\left[\bar{T}_I\ (^oK) = 0.55\ (\bar{T}_I\ (^oF) - 32) + 273\right] \qquad (1\text{-}4)$$

and P_{IM} is converted to P_{IA} (absolute). For a H_2O vacuum gauge:

$$P_{IA} = P\ (\text{in. Hg}) + \frac{P_{IM}}{13.6} \qquad (1\text{-}5)$$

3) Convert P_{IA} (in. Hg) to P_{IA} (atm.):

$$P_{IA}\ (\text{atm.}) = \frac{P_{IA}\ (\text{in. Hg})}{29.92} \qquad (1\text{-}6)$$

4) Determine pressure at outlet of each stage, P_s, atm. For stages 1, 2 and 3, $P_s = P_{IA}$ (atm.)

For stage 4,

$$P_4 = P_{IA} - \frac{(0.781)\Delta P_c}{29.92} \qquad (1\text{-}7)$$

For stage 5,

$$P_5 = P_{IA} - \frac{\Delta P_c}{29.92} \qquad (1\text{-}8)$$

5) Calculate the density of gas at inlet sampling conditions (g/cc), ρ_I:

$$\rho_I = (1.214 \times 10^{-2}) \left[\frac{P_{IA}\ (\text{atm.})\ MW_2}{\bar{T}_{I_{in}}\ (^{o}K)} \right] \qquad (1\text{-}9)$$

Where $\bar{T}_{I_{in}}$ is the average inlet temperature to the impactor.

6) Determine ρ_s, the density of the gas at the outlet of the various stages using equation 1-10

$$\rho_s = \rho_I \frac{P_s\ (\text{atm.})}{P_{IA}\ (\text{atm.})} \qquad (1\text{-}10)$$

7) Determine D_s for each stage from equation 1-11

$$D_s = \frac{-15.3\mu}{\sqrt{g_c\ \rho_s\ P_s}} + \sqrt{\frac{234\mu^2}{g_c\ \rho_s\ P_s} + \frac{2.05 \times 10^{+8}\ \mu D_c^{\ 3}\ P_s}{\rho_p\ F\ P_{IA}}} \qquad (1\text{-}11)$$

8) Determine the ratio:

$$\frac{D_5 \times 10^{-4}}{L}$$

for stage 5. L, the mean free path of gas molecules, may be determined by the following equations:

$$L = \frac{2\mu}{\rho_5\ V} \qquad (1\text{-}12)$$

$$\bar{v} = \sqrt{\frac{8\ g_c\ P_5 \times 1.013 \times 10^6}{\rho_5\ \pi}} \qquad (1\text{-}13)$$

If this ratio:

$$\frac{D_5 \times 10^{-4}}{L}$$

is greater than or equal to 2.7, equation 1-11 for D_5, the characteristic diameter, microns, as determined in step (5) is valid. The expression:

$$\frac{D_5 \times 10^{-4}}{L}$$

must be greater than or equal to 2.7 for (1-11) to be valid. This ratio is smallest for Stage 5 and increases for preceding stages. Thus, in step (8), the ratio, if satisfactory for Stage 5, is also valid for Stages 1, 2, 3, and 4. Therefore, if

$$\frac{D_5 \times 10^{-4}}{L}$$

is less than 2.7 for Stage 5, the ratio must be evaluated for Stage 4, then 3, etc., until the ratio is equal to or greater than 2.7. It should be noted that L is not the same at each stage. For those stages where

$$\frac{D_s \times 10^{-4}}{L} < 2.7,$$

D_s may be determined by equations 1-14 and 1-15.

$$D_s = 1.43 \times 10^4 \left(\frac{\mu D_c{}^3 \, P_s}{\rho_p F \, P_I C} \right)^{1/2} \qquad (1\text{-}14)$$

$$C = 1 + \frac{2L}{D_s \times 10^{-4}} \left[1.23 + 0.41 e^{-0.44 \left(\frac{D_s \times 10^{-4}}{L} \right)} \right] \qquad (1\text{-}15)$$

Although these equations may be solved explicitly, it is simpler to use a trial and error solution. To solve by trial and error, first calculate a C using the D_2 obtained from the equation 1-11. Then substitute this C in equation 1-14 and calculate a new D_s. Then calculate a new C using the last calculated value for D_s, from this C calculate another D_s. Compare the last two D_s's. If they are within 1% of each other, take the last value as D_s. If they are not, continue the procedure of calculating a C and then a D_s until 1% agreement is obtained.

9) Express the quantities collected in the cyclone, Stages 1-5, and filter as percentages of the total amount recovered. Call these r_1, r_2, etc.

10) Calculate the cumulative percentage, Σ, smaller than D_s for each stage. These are:

Filter $\Sigma_7 = r_7$

Stage 5 $\Sigma_6 = \Sigma_7 + r_6$

Stage 4 $\Sigma_5 = \Sigma_6 + r_5$

Stage 3 $\Sigma_4 = \Sigma_5 + r_4$

Stage 2 $\Sigma_3 = \Sigma_4 + r_3$

Stage 1 $\Sigma_2 = \Sigma_3 + r_2$

Brink Cyclone $\Sigma_1 = \Sigma_2 + r_1$

11) On log probability paper, plot the cumulative percentages determined in step (9) against D_s, D_s on the log scale (obtain the D_s for cyclones from manufacturer's literature).

2. PROCEDURE FOR SAMPLING THE OUTLET OF A FLUE GAS DESULFURIZATION (FGD) UNIT USING A MRI IMPACTOR

This method is applicable for determining aerodynamic size distribution of dry solid particles emitted from the TVA Shawnee flue gas desulfurization (FGD) processes at a mass loading of $0.07 g/m^3$ (0.03 gr/cfm).

The Meteorology Research Inc. (MRI) Inertial Cascade Impactor is designed to measure the aerodynamic size distribution between 0.3 and 30 μm suspended in industrial gas streams at temperatures up to 200°C (392°F) at a flow rate of 2.4 to 22.7 Lpm (0.1 to 0.8 cfm).

2.1 DOCUMENTS

2-1	Federal Register. 36(247): 24888-9.
2-2	Meteorology Research Inc. Instruction Manual for Operation, Installation, and Maintenance for the Inertial Cascade Impactor Model 1502.
2-3	Harris, D. B. Procedures for Cascade Impactor Calibration and Operation in Process Streams. EPA-600/2-77-004, January, 1977.

2.2 SAMPLING EQUIPMENT

The MRI Impactor (Model #1503) provides a total of seven cut-off stages for particulate size determination. The impactor has a collection disc located below each of the six stages as shown in Figures 10, 11, and 12. Each stage has a sequentially decreasing orifice size until the final, seventh stage which consists of a filter. The stainless steel collection plates are doughnut shaped and weigh ∿700 mg. The impactor is 2-3/4 inches in diameter by 11-1/2 inches long with a 1/2-inch NPT pipe fitting in the outlet section. Both the housing and collection plates are constructed of stainless steel with Teflon or Viton seals.

2.2.1 Impactors

MRI Model 1503 Cascade Impactor contains six impaction stages with a seventh stage using a glass fiber filter for total particulate sampling including:

- Light-weight stainless steel collection discs (MRI, Altadena, CA)
- Type A 47 mm glassfiber filters (Reeve-Angel 934-AH).

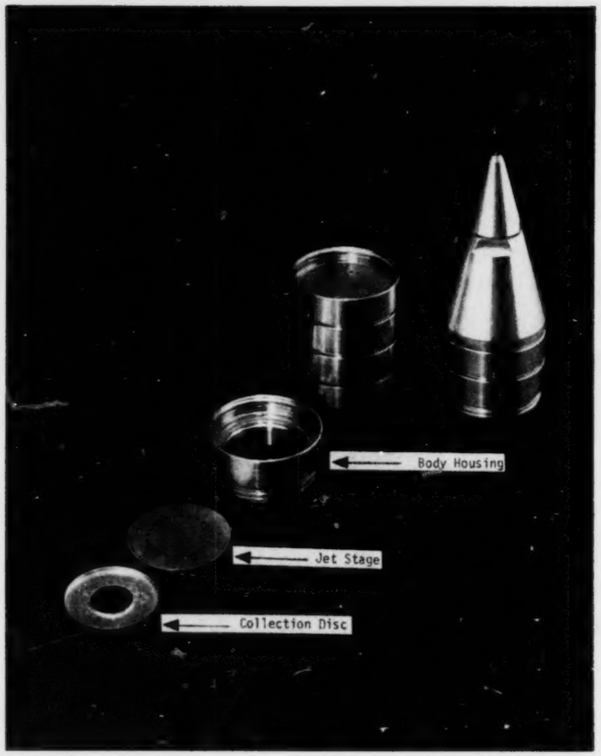

Body Housing
Jet Stage
Collection Disc

Figure 10. Upper Stages
of MRI Impactor

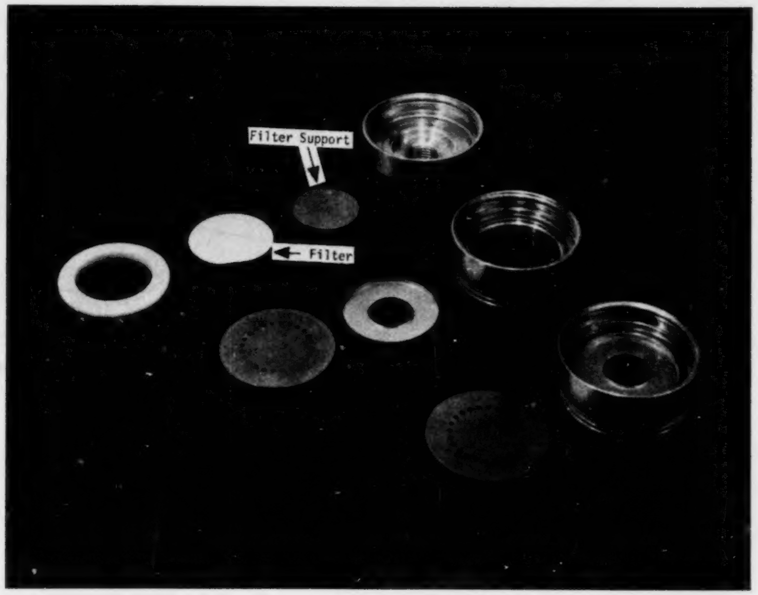

Filter Support
Filter

Figure 11. Lower Stages
of MRI Impactor

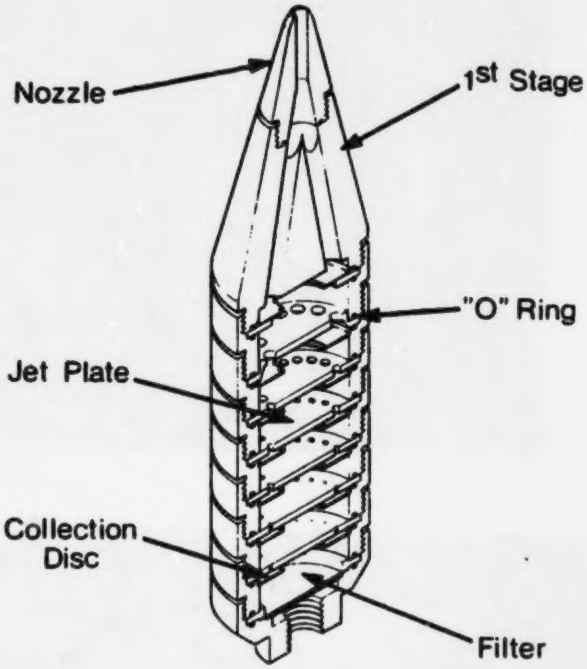

Nozzle
1st Stage
"O" Ring
Jet Plate
Collection Disc
Filter

Figure 12. Assembly Drawing of Model 1503 Inertial Cascade Impactor

- Heating Mantle for MRI Impactor (Special order from Glass-Col Apparatus Co., Terre Haute, IN).
- Out of stack 1/2" NPT connector (MRI, 464 W. Woodbury Road, Altadena, CA).

2.2.2 Sampling Probe

Aerotherm 1/2-inch OD probe with a range of nozzle sizes.

2.2.3 Aerotherm Sampling Train

- Four impingers.
- Ice bath container for impingers.
- Vacuum pump capable of pulling 4 CFM of free air and a vacuum of 12 inches of mercury or more.
- Dry gas meter.
- Flow monitoring orifice.

2.2.4 Tools and Equipment

- Spanner wrenches supplied with impactor.
- Pipe wrenches and/or clamping pliers.
- Gloves.
- Teflon pipe fitting tape.
- Stopwatch.
- Isokinetic Flowrate Calculator (Aerotherm Corp., Model #HVSS-901).
- A source of 110V electrical power must be provided at the sampling location.
- Suitable platforms must be provided at the sampling location for placing Aerotherm alongside the sampling port and for working space for the operator.
- Five-place analytical balance.

2.2.5 Equipment and Materials for Coating Collection Discs

- Grease, Apiezon H grease.

- Drierite, 5 lb.
- Large desiccator, Kimble #21050, 250 mm min. diameter with porcelain plate.
- Petri dishes (top and bottom), 60x15 mm pyrex.
- Forceps.
- PVC gloves, U.S. Industrial Gloves, Compton, CA.
- Kimwipes.
- Acetone, reagent grade.
- Dow 111 high vacuum grease.

2.2.6 Probe Connection

- Two Swagelok Quick Disconnect SS-QF8-B-810-VT.
- Two Swagelok Quick Disconnect SS-QF8-S-810.

2.3 REQUIREMENTS

2.3.1 System Design

The MRI system is operated out of stack using the 1/2" Aerotherm probe to extract sample from the flue gas and the Aerotherm impinger, pump, and the control unit to measure the gas flowrate. By placing the impactor out of stack, a larger capacity heating mantle can be used. The higher temperatures attainable with this mantle will prevent premature collection of H_2SO_4 due to condensation on the first several stages.

2.3.2 Sampling Procedure

The flow rate through the impactor will determine the size cut-offs that each stage will collect. As will be described in Section 2.4, an average isokinetic sampling rate will be determined. Once the flow rates are established, they must be maintained throughout the run regardless of the individual velocities at each point.

2.3.3 Handling of Collection Discs

Care must be taken to limit contact with the discs. At no time should the discs be touched with ungloved hands. All laboratory manipulations are to be performed in a clean environment using tweezers to handle the discs.

It is important to remember that several grains of dust could represent the total weight captured on a disc. Contamination control is essential during greasing, drying, and weighing.

2.3.4 Calibration and Maintenance

After each run, the probe nozzle, probe, connecting lines, S-pitot tubes, impactor, and impinger system must be cleaned. The probe nozzle, probe, and connecting lines can be cleaned with a long handle test tube brush and backflushed with high pressure air. Should further cleaning be required, deionized water followed by acetone (or isopropyl alcohol) can be used. The S-pitot tube should be backflushed with a high pressure air line. The impactor cleaning procedures are detailed in Chapter 4. The impinger system is flushed out and the proper solvents replaced in the impinger bottles prior to the next run.

In addition to these procedures, the C_p of the S-pitot and the $\Delta H@$ of the flowmeter orifice are determined every two months. If any evidence of corrosion appears (pitting, scale build-up, etc.), the C_p and $\Delta H@$ recalibration procedure should be repeated as needed. Tables 7 and 8 in Chapter 4 contain a summary of recommended maintenance and troubleshooting procedures.

2.3.5 Cleanliness

Gas carrying lines should be cleaned weekly. No particulate build-up in the pitot tube can be tolerated. Impactors must be cleaned completely after use. Chapter 4 describes the maintenance schedule for the sampling and analysis equipment.

2.3.6 Safety

OSHA safety requirements with regard to working environment and operator safety will be met at all times. The reagents mentioned in the procedure are not extremely toxic but can be harmful if misused.

2.4 PROCEDURE

The MRI impactor system consists of a 1/2-inch Aerotherm probe connected directly to the impactor (Figure 13). The MRI system is used as an out-of-stack extractive sizing method. Using an Aerotherm probe, a

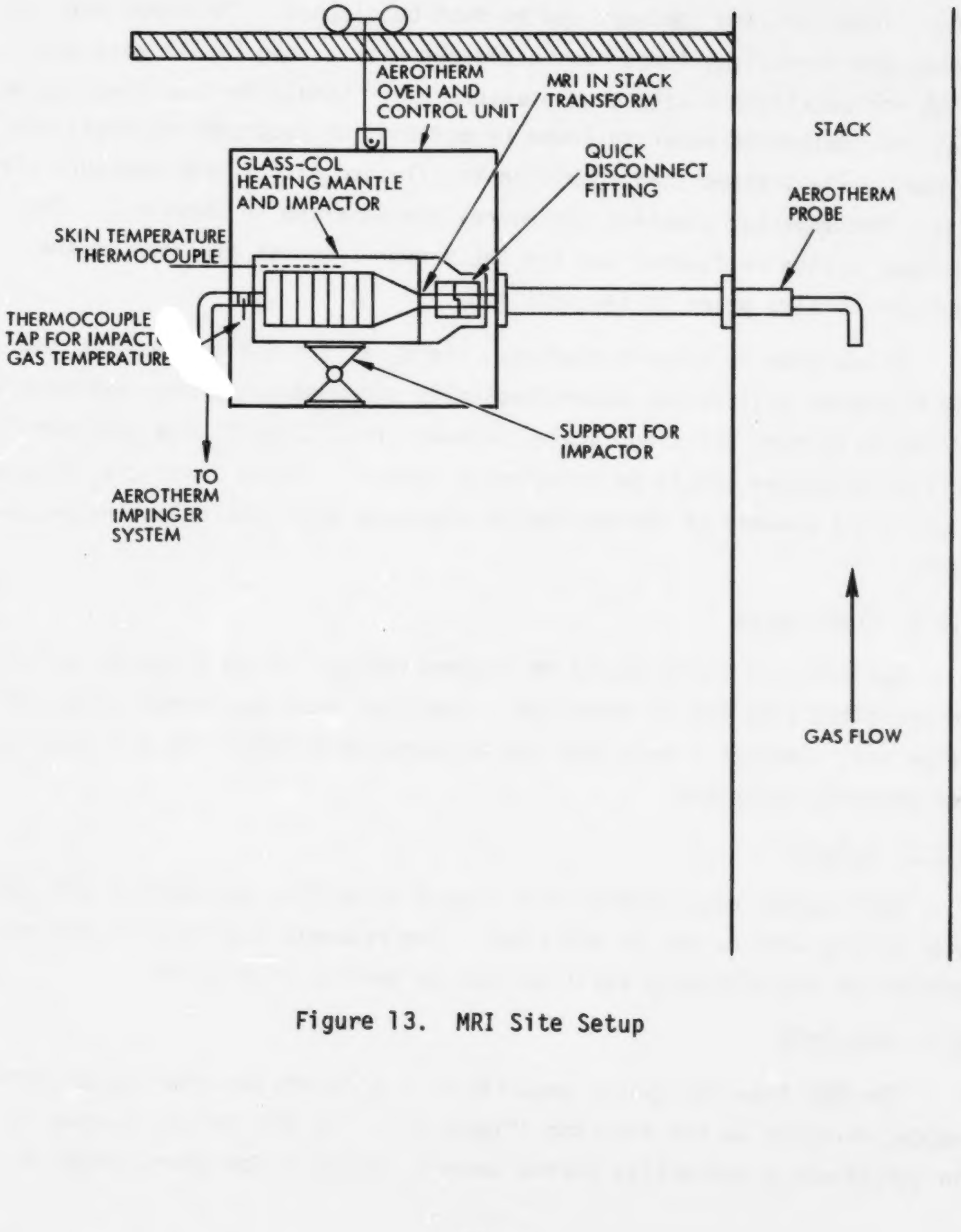

AEROTHERM OVEN AND CONTROL UNIT
MRI IN STACK TRANSFORM
STACK
QUICK DISCONNECT FITTING
GLASS-COL HEATING MANTLE AND IMPACTOR
AEROTHERM PROBE
SKIN TEMPERATURE THERMOCOUPLE
THERMOCOUPLE TAP FOR IMPACT GAS TEMPERATUR
SUPPORT FOR IMPACTOR
TO AEROTHERM IMPINGER SYSTEM
GAS FLOW

Figure 13. MRI Site Setup

velocity profile for the duct is obtained. The average velocity is calculated and used to select a nozzle that will sample at the average isokinetic velocity, but less than 22.6 lpm (0.8 acfm).

Temperature control of the impactor system is maintained by monitoring the stack and outlet gas temperature from the impactor. The necessary heat is supplied by a specially designed Glass-Col heating mantle. The gas flow rate is monitored by measuring the ΔH across a calibrated orifice with a magnehelic gauge.

The amount of material collected is determined by weighing the collection stages before and after the run. Particulate matter collected during the probe and tubing rinses is added to the weight of the first stage. The collection plate and filter are thermally conditioned and desiccated prior to weighing. After sampling, the samples are desiccated to constant moisture content prior to reweighing. Because of the potential for systematic errors in weighing, blanks consisting of spare collection stages and filters are conditioned and weighed along with the samples to monitor weighing errors. If weight changes greater than 0.1 mg occur in the blanks, the weight gain or loss is subtracted or added, respectively, to the weight of the samples (see Section 4.1.8 for further details).

NOTE

> The critical checklists (Table 5) for the MRI should be available to the personnel performing the test run. This checklist will provide an excellent guideline for the sampling site and laboratory personnel.

2.4.1 Laboratory Preparation of MRI Cascade Impactor

a) Disassemble the MRI impactor by unscrewing each stage. Figure 12 shows an assembled unit. Inspect the jet plates prior to assembly to ensure that clogging has not occurred. Clean each collection disc and jet plate by wiping the surface with a Kimwipe wetted with acetone. Inspect the disc and plate after cleaning for particulate or finger marks on the collection disc and jet plate. Inspect interior of the impactor housing for particulate matter. Clean the interior of the impactor after removing the Viton O-rings with a squeeze bottle containing acetone and Kimwipes.

For hard to reach areas, use a camel-hair brush to remove the particulate. The threads on the impactor should be lightly greased with Dow 111 high vacuum grease.

b) With the tweezers, dip the lightweight SS collection disc into a 250 mL beaker containing 200 mL of reagent grade toluene to clean the surface. Withdraw the disc and hold it in air until the toluene has dried. Place the discs in separate labeled petri dishes.

c) Using a rubber policeman, spread a thin coating of Apiezon H around the center of the doughnut-shaped collection disc. Should some of the Apiezon H be spread over the edge of the disc, clean the disc with toluene and repeat the procedure at 2.4.1.(b). Prepare eight discs, six for use and two as spares.

NOTE

> At all times these manipulations are to be performed in a dust-free environment.

d) Place the covered petri dishes with the eight collection discs into an oven for 4 hours at 175°C (347°F). The filters are heated for 4 hours at 287°C (550°F).

e) After 4 hours, remove the petri dishes with discs and filter, and allow them to equilibrate in the desiccator for 2 hours.

f) Once the discs and filters have been dried and desiccated, they are weighed on a balance capable of weighing to the nearest 0.01 mg. Remove the petri dishes and filters from the desiccator just prior to weighing (keep desiccator closed otherwise). Remove the discs from petri dish with a forceps being careful not to touch the greased area and place them on the balance. After weighing, record the weight and disc number on the Laboratory Data Sheet (Figure 14). Place the filters and the coated discs back in the petri dishes, cover them, and place the petri dishes near the impactor.

g) Using forceps, place a preconditioned 47 mm diameter glass fiber filter on top of the filter support housing. Then place the locking spacer on top of the filter.

MRI - DRY AEROSOL SIZE DISTRIBUTION
LABORATORY DATA SHEET

SAMPLE LOCATION ____________

DATE/TIME ____________

RUN NUMBER ____________

DATE		WEIGHT			%	% CUM	MICRONS d_{50}
STAGE	DISC*	FINAL	TARE	GAIN			
FILTER							
TOTAL							

% - Weight gain on each stage divided by the total weight gain.

CUM% - Starting with the filter accumulate each stage to arrive at the cumulative percent smaller than the previous d_{50}.

* - Disc Code for labeling petri dishes should be the date of run, stage no. and run letter series (example: 8/27/75, 1A; 8/27/75, 2A; etc.). The letters series represents the sequencial number for each successive run that day. 8/27/75, 1A; 8/27/75, 1B would be the next run.

Figure 14. MRI Laboratory Data Sheet

h) Screw the body housing into position to receive the collection disc, and replace the Viton O-ring. Using forceps, insert collection disc firmly into the housing groove. Place jet plate on top of disc into groove. Continue until all stages have been connected and the impactor is completely assembled as per assembly drawing in Figure 12.

i) After the MRI impactor is assembled, it should be leak checked in the laboratory. Connect a vacuum gauge to the inlet of the impactor, and attach the outlet of the impactor to the in-house vacuum line.

j) Leave the vacuum on until the gauge indicates 380 torr (15 in. Hg).

k) Close the vacuum line and note any rise in pressure. The vacuum should not vary over several minutes.

l) If a leak is noted by a decreasing vacuum reading, check the impactor to verify that all connections are tight and the vacuum gauge is working. Be sure that all the vacuum lines have tight seal as well. If these measures do not locate the leak, take the impactor apart and replace any suspicious O-rings, then repeat the vacuum test.

m) Once the impactor is leak checked, both ends are sealed to prevent dust from entering and the impactor is taken to the sampling site.

2.4.2 Measurements and Calculations for Isokinetic Sampling

a) The duct geometry must first be considered. For circular 40-inch diameter ducts as encountered on the Shawnee limestone wet scrubber, refer to Figure 6.

b) Sixteen sample points are selected, eight are along one axis across the duct, and eight lie along another axis at 90^o to the first. The location of each sample point is obtained from Table 1.

c) The test site should consist of a sampling port in the stack with an opening to allow the easy insertion of the sampling probe. It should be sealed to minimize the disturbance of the flow during sampling and protect personnel and equipment from hot exhaust gases. Also, the test site must have a platform to provide for the safety of personnel and equipment. The electrical power required to operate the equipment is approximately 35 amp/115V.

2.4.3 Isokinetic Sampling with the Aerotherm System. The Aerotherm sampler is capable of isokinetic sampling if the nozzle inlet velocity matches the exhaust stack velocity when a sample is taken. The control unit contains a set of gauges to measure the pitot pressure (stack velocity) and the pressure difference across an orifice (sampling rate). By adjusting the control valve on the pump, the flow rate can be varied thereby changing the inlet velocity at the nozzle.

Prior to the initiation of the sizing program, the Aerotherm S-pitot and flow orifice are to be recalibrated. The C_p and $\Delta H@$ are measured and entered into the MRI Field Data Sheet (FDS) (Figure 15).

a) Using the S-pitot attached to Aerotherm probe, perform a sixteen point two-diameter velocity traverse across the duct. Refer to Table 1 for the position of the sampling points. At each point, record the ΔP, $\sqrt{\Delta P}$ and the stack temperature (T_S) on the Field Data Sheet.

b) Determine the average stack temperature ($\overline{T}_S$) and the average $\sqrt{\Delta P}$ in inches of H_2O. Record these values on the ΔP and T_S table on the Field Data Sheet.

c) Using the Isokinetic Flow Rate Calculation, complete Table 2 on the FDS.

NOTE

For detailed instructions on how to use the Isokinetic Flowrate Calculator, see Appendix A.

The following are the condensed instructions found on the back of the calculators:

1) Set C_p at $\Delta H@$.

2) Using hairline, set % H_2O at arrow.

3) Read index number at arrow.

4) Set T_m at index number.

5) Read second index number at T_S.

6) Set P_S/P_m at second index number. If the Nozzle Size (D_n) is known, proceed to Step 9. If not, proceed to Step 7.

7) Set the average $(\sqrt{\overline{\Delta P}})^2$ to Reference Arrow C on ΔH scale.

8) Read exact Nozzle Size at Reference Arrow B on D_n scale. Select available nozzle that is near this diameter and suitable for use.

9) Set Nozzle Size (D_n) under Reference Arrow B.

10) Read ΔH setting opposite $(\sqrt{\overline{\Delta P}})^2$ reading using Cursor as needed.

11) Record the ΔH as needed on Column 2 in the work sheet.

12) Reset Hairline over C_p.

13) Set $\overline{T}_S$ at Hairline and move Hairline over V1 arrow.

14) Set V2 Arrow under Hairline and move Hairline over ΔP.

15) Turn over calculator without moving Cursor and set M_S under Hairline.

16) Read Stack Velocity (V_S) at Stack Pressure (P_S).

17) Record all these data on Table 2 on the FDS.

1. ΔP AND T_S TRAVERSE DATA

SAMPLE PORT	TRAVERSE POINT	ΔP in. H_2O	$\sqrt{\Delta P}$	T_S, °F
			$(\sqrt{\Delta P})$ AVG. =	$\bar{T}_S$ =
	AVERAGE			

2. DATA OF NOZZLE SIZE, ΔH, AND V_S CALCULATIONS

VARIABLE	VALUE
$\Delta H_@$	
C_p	
% H_2O	
T_m	
$\bar{T}_S$	
P_m	
P_S	
D_n	
$(\sqrt{\Delta P})^2$ AVG.	
ΔH	
M_S	28.6
V_S	

Figure 15. MRI Field Data Sheet

-Continued-

FIELD DATA SHEET

DATE/TIME ______________________
RUN NUMBER ______________________
OPERATOR ______________________
AMBIENT TEMPERATURE ______________________
BAROMETRIC PRESSURE ______________________
STACK PRESSURE, (P_S) ______________________
INLET GAS FLOW ______________________
REHEATER AIR FLOW ______________________
P_S/P_m ______________________
LEAK RATE ______________________

SCHEMATIC OF TRAVERSE POINT LAYOUT
READ AND RECORD ALL DATA EVERY______MINUTES

TRAVERSE POINT NUMBER	CLOCK TIME (24 HOUR CLOCK)	GAS METER READING (CUBIC FEET)	ΔP	ORIFICE PRESSURE DIFFERENTIAL (ΔH, IN. H_2O)		STACK TEMPERATURE (T_S), °F	DRY GAS METER TEMPERATURE		PUMP VACUUM IN. Hg	IMPACTOR TEMPERATURE, °F	
				DESIRED	ACTUAL		INLET $(T_{m_{in.}})$, °F	OUTLET $(T_{m_{out}})$, °F		SKIN	OUT
INITIAL											

Figure 15. MRI Field Data Sheet

d) Symbols

$\Delta H@$ = Orifice calibration coefficient (in. H_2O)

C_p = Pitot tube coefficient (unitless)

T_m = Temperature of dry gas meter (Average of T_{IN} and T_{OUT} in ^oF)

$\overline{T}_S$ = Average stack temperature (^oF)

ΔH = Orifice Pressure Drop (in. H_2O)

P_S = Stack pressure (in. H_2O)

T_S = Temperature of Stack at specific points (^oF)

ΔP = Pressure drop across pitot tube (in. H_2O)

V_S = Stack Velocity (ft/sec)

P_m = Meter Outlet pressure (in. H_2O)

D_n = Nozzle Diameter (inch)

$(\sqrt{\overline{\Delta P}})^2$ = Average pressure drop across pitot tube (in. H_2O)

2.4.4 Equipment Set-Up

a) Set-Up and Operation of Shawnee Aerotherm Sampler. The probe is mounted on the side of the sampling oven and is adjusted for height by means of a line cinch attached to the oven support bracket. Connectors and plugs are attached to the cabinet, consisting of:

- Pitot lines color coded for proper connection
- Power plug for probe heater. The receptacle is located on the side of the sampling cabinet.

The impinger bottles, two of which are filled with sodium carbonate, one left empty, and one filled with silica gel, are contained in a separate ice-cooled box. The cabinet controls consist of:

- Magnehelic gauges indicating the pitot pressure corresponding to stack velocity (V_S) and the orifice pressure (ΔH) drop.
- The multi-point temperature indicator measuring the stack temperature, (T_S), gas entering and leaving the dry gas meter, (T_m).
- Gas meter for the total volume of gas sampled.

The MRI impactor consists of six impaction stages and a back-up filter. Using the appropriate connecting tubing, the MRI impactor will be connected to the probe and mounted in the Aerotherm oven (Figure 13).

1) Using $(\overline{\sqrt{\Delta P}})^2$ and $\overline{T}_s$ calculate the correct nozzle to meet the average isokinetic conditions.

2) Attach this nozzle to the Aerotherm probe.

3) Brush and rinse inside the Aerotherm probe to verify no particulate remains.

4) Connect the impactor to the probe via a Swagelok 1/2" quick disconnect with a 1/2" male NPT fitting screwed into the MRI 1/2" female inlet connector.

5) The first two impingers are filled with saturated Na_2CO_3. The third is left empty and the fourth impinger is filled with 250 g of silica gel.

6) Once the vacuum lines are attached, the Aerotherm nozzle is plugged using a rubber stopper.

7) Open the vacuum pump valve until the gauge indicates 380 torr (15" Hg vacuum). The flow through the dry gas meter should be less than 0.02 cubic feet per minute before the sample can be taken.

8) If the leak rate is less than 0.02 cubic feet per minute, close the vacuum valve slowly to prevent a pressure surge and remove the stopper. If leak rate is greater than 0.02 cfm, tighten all fittings and examine the pump for wear. Replace worn parts and repeat leak tests. Once acceptable leak rates are met, the unit is ready for sampling.

2.4.5 Operation of Aerotherm and MRI Impactor During Sampling

Once the ΔH is determined, the Aerotherm and MRI units are ready to run. Although a sampling traverse will be performed, the units will be run at the same ΔH.

NOTE

Because the volumetric flow determines the particle size cut-offs for each impactor stage, the flow rate in the MRI impactor must remain constant throughout the run regardless of the ΔP reading at the traverse point.

a) Set the probe temperature controller and the MRI gas out temperature at $12^{o}C$ ($25^{o}F$) above the highest temperature in the stack, but not higher than $175^{o}C$ ($347^{o}F$).

b) Turn the pump on and immediately insert Aerotherm probe into the stack until it is at the first sampling point.

c) Adjust ΔH on the magnehelic gauge for the average setting previously determined in traverse. Constantly check and record on a field data sheet the pitot manometer (ΔP) reading, stack temperature (T_S), dry gas meter temperature (T_m), vacuum pump pressure, and the skin and gas out impactor temperature. Be sure to complete all other information on the data sheet.

d) Sample for 7.5 minutes at each point for a total of two hours Sampling times should be adjusted to collect a maximum of 10 mg/stage.

NOTE

The precalculated ΔH must be maintained at each point.

e) At the end of the run, the probe is removed from the stack and the Aerotherm unit is shut down. Slowly close the flow control valve to dissipate any back pressure in the system and thus prevent water from surging forward in the impinger system. Stop the stopwatch and record final gas meter reading on field data sheet.

f) Using gloves, remove the MRI impactor with heating mantle attached from the oven and return it immediately to the laboratory. Cover the top of the impactor to prevent particles from entering.

NOTE

During this period, avoid jarring the impactor. Extreme care should be taken to avoid the addition or loss of collected particulate. Carry the impactor upright and do not expose it to dust.

g) Carefully rinse the probe with reagent grade acetone collecting the rinse until a clean stream of liquid issues from the probe.

NOTE

> Take extreme care in performing this task as the small amounts of particulate matter recovered represent a large portion of the total particulate aerosol collected. Both contamination and loss of sample must be avoided. Any accidents which occur must be recorded on the field data sheet.

h) Once the impactor is transferred to laboratory, clean the outside of the impactor of any dust. This should be done in the prep room prior to entering the clean room.

i) Be sure that the correctly labeled petri dishes are nearby, ready to accept the collection plates as they are removed.

j) With the impactor in an upright position, begin to remove the housing starting at the top. Inspect the inlet nozzle for any sign, no matter how little, of particles collected on the inlet walls. Note the presence of the particulate matter on the back of the laboratory data sheet.

k) If any particles are found, they should be carefully brushed onto the collection plate below their collection point.

l) Inspect all the jet nozzle plates for any sign of pitting or corrosion. Especially inspect the underneath of the jet nozzle for particles that might have collected there. If any particles are found, note this fact along with a description of any patterns formed, color, or quantity obtained on the back of the laboratory data sheet.

m) Carefully brush these particles onto the collection plate below the jet nozzle plate.

n) Repeat these activities for all the stages and the filter. In the filter's case be sure that all fragments of the filter are removed from the filter support; even the loss of the smallest fragment can affect the weight of the filter.

o) Desiccate the collection plates and filter for 2 hours.

p) Evaporate enough of the acetone from the probe rinse so that all the particulate and the remaining acetone can be quantitatively transferred to a tared 30 mL beaker. At all times, handle the 30 mL beaker with gloved hands.

q) Evaporate the bulk of the acetone from the 30 mL beaker on a hot plate allowing the rest of the acetone to air dry in a clean dust-free area. Dry the particulate at 110°C for two hours and desicate with the rest of samples for approximately two hours.

r) Weigh the collection plates and filter to the nearest 0.01 mg. At the same time weigh the balance and sample blanks (see section 4.1.8.(a) for specific procedures to correct for any weighing errors). Record the data on the laboratory data sheet.

2.5 DATA REDUCTION

The MRI unit is designed to provide a distinct particle size cut-off at each stage. Using Figure 16 and the volume flow corrected to standard impactor conditions, the d_{50} (cutoffs) for each stage can be determined.

1) Calculate the change in weight for each collection disc, and filter.

2) Add up the differences to get the total particulate weight collected on discs and filter.

3) Divide the amount collected on each plate by the total amount collected to find what percent of the total is impacted on each plate.

4) From the field test log, determine the total volumetric gas flow in ft^3/min. Correct this flow (Q_m) to impactor conditions (Q_S):

Q_m = Sampling rate at dry test meter (ft^3/min.)

Q_S = Flow rate at impactor conditions (ft^3/min.)

B_w = Volume fraction of moisture in gas stream

T_i = Average impactor gas out temperature

$$Q_S = Q_m \left(\frac{29.92}{P_S}\right) \left(\frac{T_i + 460}{T_m + 460}\right) \left(1 + B_w\right) \tag{2-1}$$

where T_i and T_m are in ^{o}F.

5) Using Figure 16 with the gas flow rate at impactor conditions (Q_S) and the impactor temperature (T_i), determine the d_{50} for each stage.

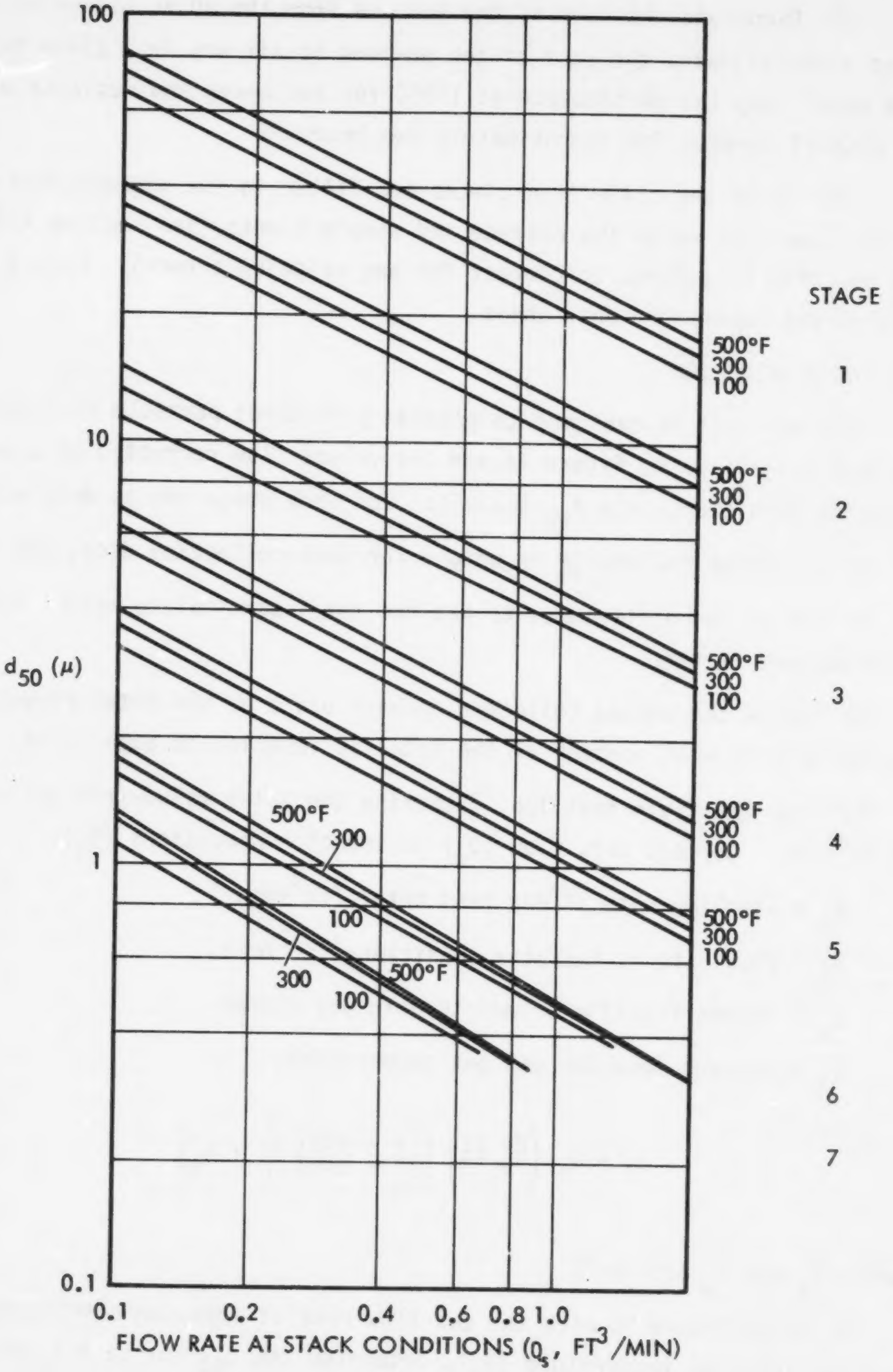


Figure 16. MRI Impactor Stage Cut-off Diameter (μ)

6) The results can be plotted on log probability paper with the particulate diameter (d_{50}) as the ordinate and cumulative percent by weight as the abscissa.

7) The cumulative weight percent for Stage 1 is determined by subtracting the weight percentage for stage 1 from 100. For stage 2, the cumulative weight percentage is found by subtracting the weight percentages from stages 1 and 2 from 100. This process is repeated for all the stages.

8) An alternate approach is to normalize the data. This approach $\left[(dm/d(\log D_{50})\right]$ is not discussed here, but is detailed in Reference 2.3 and is the recommended approach if the Brink and the MRI data are to be compared.

9) The mass loading is found by correcting the volume of air passed through the dry gas meter to STP (A_{STD}):

$$A_{STD} = (Q_m)\ (120\ \text{min.}) \left(\frac{492}{T_m + 460}\right) \left(\frac{P_S}{29.92}\right) \qquad (2\text{-}2)$$

$$\text{Mass Loading (mg/dcfm)} = \left(\frac{\text{Total weight found on stages and filter}}{A_{STD}}\right) \qquad (2\text{-}3)$$

where T_m is in °F and P_S is in inches of Hg.

3. DETERMINATION OF H_2SO_4 VAPOR USING A CONTROLLED CONDENSATION COIL

This method was designed to measure the vapor phase concentration of SO_3 as H_2SO_4 entering the flue gas desulfurization until(FGD) and exiting from the reheater at the TVA Shawnee Power Plant in Paducah, Kentucky. This method is specifically designed to operate at temperatures up to 250°C (500°F), 3000 ppm SO_2 and 8-16% H_2O. By using a modified Graham condenser, the gas is cooled to the acid dew point at which the SO_3 (H_2SO_4 vapor) condenses. The temperature of the gas is kept above the water dew point to prevent an interference from SO_2 while a heated quartz filter system removes particulate matter. The condensed acid is then titrated with 0.02 N NaOH using bromophenol blue as the indicator.

3.1 DOCUMENTS

3-1 Federal Register. 36(247): 24888-9.

3-2 Goksoyr, H. and K. Ross, J. Inst. Fuels, 35, 177 (1962)

3-3 Lisle, F.S. and J.D. Sensenbaugh, Combustion, 1, 12 (1965).

3-4 Nacovsky, W., Combustion, 1, 35 (1967).

3-5 Standard Methods for the Examination of Water and Wastewater, 13 Edition, pages 52-56 (1971).

3-6 Maddalone R., C. Zee, and A. Grant, "Procedure for Titrimetric Determination of Sulfate Using Sulfonazo III Indicator," TRW Systems, EPA Contract No. 68-02-1412, Task 6, Feb. 14, 1975.

3.2 EQUIPMENT AND MATERIALS

3.2.1 Sampling Materials

- Probe construction materials (including materials for two 3-foot probes and spare).

  a) Three Vycor tubes 0.5-inch OD x 36-inch with a 18/9 female ball-and-socket joint placed on one end (special order -- A. H. Thomas or Ace Glass, see Figure 17).

  b) Three glass insulated heating tapes - 1/2-inch x 72-inch; 288 watts (Fisher Sci. Co. #11-463-50C or equivalent).

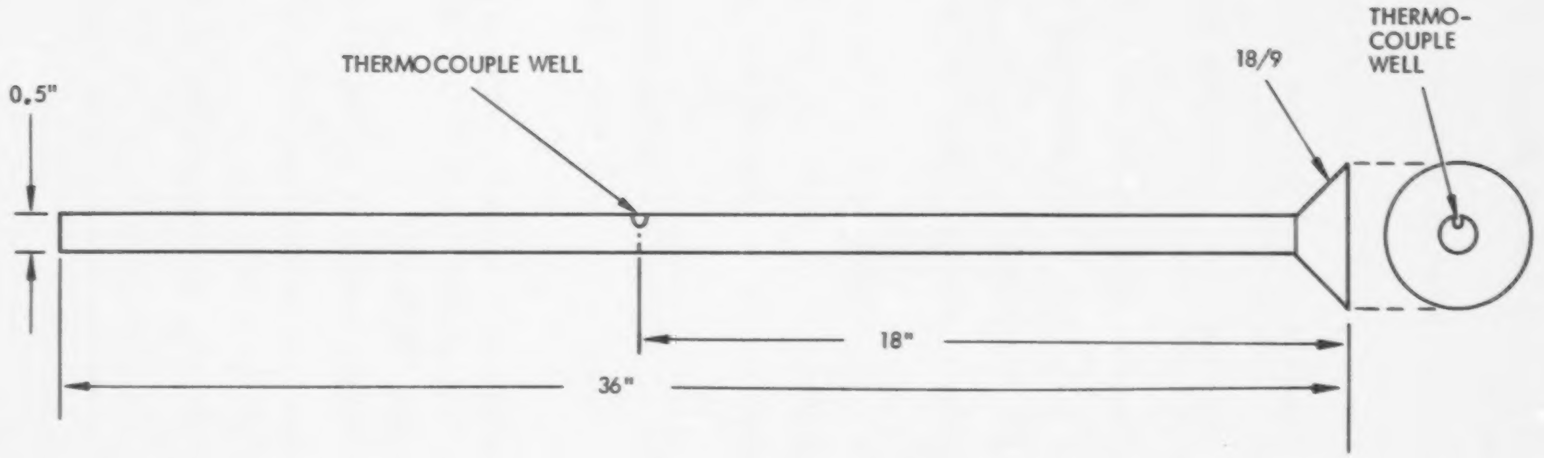

THERMOCOUPLE WELL
0.5"
18/9
THERMO-
COUPLE
WELL
18"
36"

Figure 17. Vycor Sampling Liner

c) Three 33-inch x 1-inch x 0.065 inch wall 304 SS tubes used as probe sheaths.

d) One dozen silicone rubber No. 6 stoppers (A.H. Thomas #8747-E65).

e) Glass tape (Scotch glassfiber electrical tape).

f) Four Omega (Stanford, Conn.) shielded thermocouples (I/C), (TH36-ICSS-18G-12) with 8-foot lead.

g) Four Omega (Stanford, Conn.) unshielded thermocouples (I/C), (IRCO-032 with 8-foot lead).

h) Six Omega male connectors (ST-IRCO-M).

i) Two six-foot heavy duty (∿20A) electrical cords.

j) Two 1-1/2 inch hose clamps.

k) Two square yards of asbestos cloth (VWR, Atlanta, Georgia, #10930-009).

l) Three adaptors for connecting hoses (Ace Glass, #5216-23).

m) One Teflon Swagelok Union (T-810-6).

- Two pumps capable of pulling 1 cfm of free air (Brink impactor pump may be used).

- Bath controller-circulator (A.H. Thomas #9840-B15 or equivalent).

- Fifty feet of 1/2-inch x 1/4-inch rubber tubing (A.H. Thomas, #9544-R57).

- Three Graham condensors (controlled condensation coils - CCC) modified to hold an enclosed 60 mm medium frit (special order from Ace Glass, Louisville, Ky.; see Figure 18).

- Two styrofoam chests capable of holding a 2-gal. bucket.

- Three glass insulated heating tapes, 3/8-inch x 24-inch, 96 watts (A.H. Thomas, #5954-H22 or equivalent).

- Four autotransformers, variable, 10 amp. (A.H. Thomas #9461-D10 or equivalent).

- One hundred Tissuequartz filters, 37 mm diameter (Pallflex Corp. Kennedy Drive, Putnam, Conn. 06260).

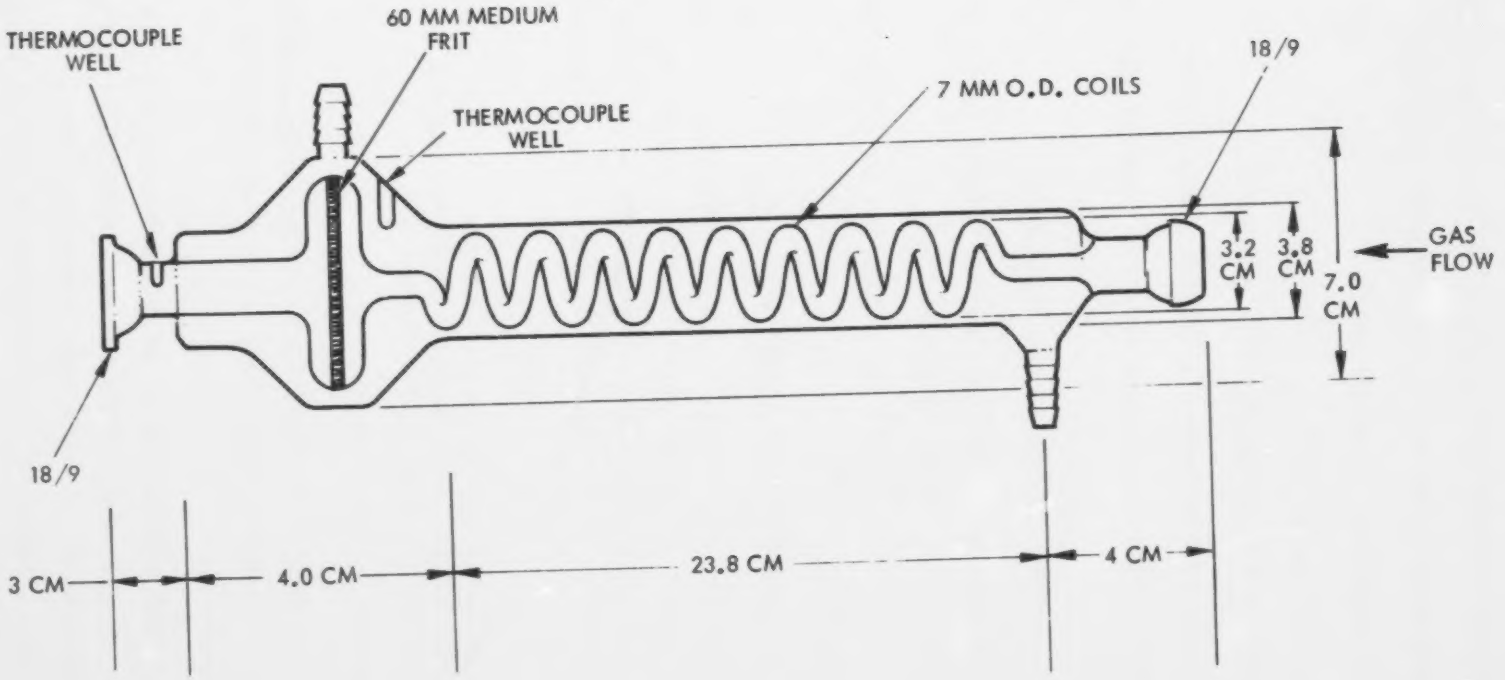

THERMOCOUPLE WELL
60 MM MEDIUM FRIT
THERMOCOUPLE WELL
7 MM O.D. COILS
18/9
3.2 CM
3.8 CM
7.0 CM
GAS FLOW
18/9
3 CM
4.0 CM
23.8 CM
4 CM

Figure 18. Controlled Condensation Coil

- Eight pinch clamps (A.H. Thomas 2841-21 or equivalent).
- Three Greenburg-Smith type impingers or equivalent.
- Sodium carbonate, technical grade.
- Indicating silica gel.
- Stopcock grease (Ace Glass Co., #8229-10).
- Three-inch bushing with a 1-1/8 inch hole drilled in the center.
- Two RdF digital temperature indicators-series-2000 with iron/constantan sensors.
- One vacuum gauge (A.H. Thomas #5654-B10).
- Two soap bubble flowmeters (Applied Science Laboratory, P.O. Box 440, State College, Penn. 16801, (814)-238-2406.
- Glass-Col heating mantle for filter system (Glass-Col, 711 Hulman St., Terre Haute, Ind.).

3.2.2 Reagents and Apparatus for H_2SO_4 Titration

- Carbon dioxide-free distilled water - Prepare all stock and standard solutions, and dilution water for standardization procedure, using distilled water which has a pH of not less than 6.0. If the water has a lower pH, it should be freshly boiled for 15 minutes and cooled to room temperature.

NOTE

> Deionized water may be substituted for distilled water provided that it has a conductance of less than 2 microohms/cm and a pH greater than 6.0.

- NaOH pellets - reagent grade.
- Stock 1.0 N NaOH - Dissolve 40 g of reagent grade NaOH in 1 liter of CO_2 free distilled water. Store in a pyrex glass container with a tight fitting rubber stopper.
- 0.0200 N NaOH - Dilute 20 mL of 1 N NaOH with CO_2 free distilled water to 1 liter. Store in a tightly rubber stoppered pyrex glass bottle protected from atmospheric CO_2 by a soda lime tube. For best results, prepare daily. This solution will be standardized against potassium biphthalate (see Section 3.4.3.(b))

- Potassium biphthalate ($KHC_8H_4O_4$)-Anhydrous, reagent grade.

- 0.0200 N potassium biphthalate (KHP) solution - Dissolve 4.085 g of dry (110°C for 1 hour) KHP into 1 liter of CO_2 free distilled water.

NOTE

- The normality of the KHP solution equals (wt. KHP)/204.2.

- Anhydrous ethyl alcohol - U.S.P. or equivalent.

- Phenolphthalein indicator solution - Dissolve 0.05 g of reagent grade phenolphthalein in 50 mL ethyl alcohol and dilute to 100 mL with CO_2 free water.

- Bromophenol blue indicator solution - Dissolve 0.1 g in 7.5 mL of 0.02 N NaOH. Dilute to 250 mL with CO_2 free distilled water.

- Ten milliliter micro-buret, Kimble 17132F (A.H. Thomas #1993-M-30 or equivalent).

- Desiccator (A.H. Thomas #3751-H10 with cover and plate to fit).

- Drierite desiccant - 5 lb. Dierite (A.H. Thomas #C288-T49).

- Four Erlenmeyer flasks with 28/15 ball and socket joint, 125 mL (Ace Glass Co., Louisville, Ky., #6975 or equivalent).

- Four stoppers for 28/15 ball and socket joint (Ace Glass Co., #8263-08 or equivalent).

- Four 50 mL volumetric flasks.

- Dowex 50W-X8 cation exchange resin 20 to 50 mesh.

- Barium perchlorate trihydrate, reagent grade.

- 0.01 M $Ba(ClO_4)_2 \cdot 3\ H_2O$ - Transfer approximately 3.9 of reagent grade barium perchlorate trihydrate into a one liter reagent bottle. Add enough D.I. H_2O to dissolve the sale and then dilute to the mark.

- Sulfonazo III Solution, 0.1% W/V - Transfer 0.025 g of sulfonazo III into a 25 mL bottle, add water to dissolve the indicator and fill to the mark.

3.3 REQUIREMENTS

3.3.1 System Design

The SO_3 (H_2SO_4 vapor) Controlled Condensation System (CCS) consists of a heated Vycor probe, a modified Graham condensor (condensation coil), a critical orifice, impingers, and pump (see Figure 19).

3.3.2 Sampling

Since a gas, SO_3 (H_2SO_4 vapor), is being sampled, no traverse will be performed in the stack. The sample probe will be positioned at a point representative of the stack flow.

Flow control in the CCS is maintained by monitoring the dry test meter with a stopwatch.

3.3.3 Handling of Glassware

Because of the corrosive nature of SO_3 (H_2SO_4 vapor), only Vycor and Pyrex glassware is used. Severe mechanical shocks are to be avoided, especially when the probe is heated to 250°C (500°F). Never place any strain on glass ball joints and clean the ball joints of grease and dirt after each run.

3.3.4 Calibration and Maintenance

After each run the probe, connecting lines, controlled condensation coil, filter holder, and impinger system must be cleaned. The probe and connecting lines can be cleaned with a long handle test tube brush and backflushed with high pressure air. If particulate matter adheres to the inside of the probe, rinse with deionized water followed by acetone (or isopropyl alcohol). The impinger system is flushed out and the proper solvents are then replaced in the impinger bottles prior to the next run. The filter holder is inspected and cleaned before the next run and the filter pad is replaced. Table 8 in Chapter 4 details the recommended maintenance for the CCS by component.

3.3.5 Cleanliness

Contamination of the condensation coil rinse solutions must be avoided to prevent neutralization of the H_2SO_4. Keep the rinse solutions in a covered flask.

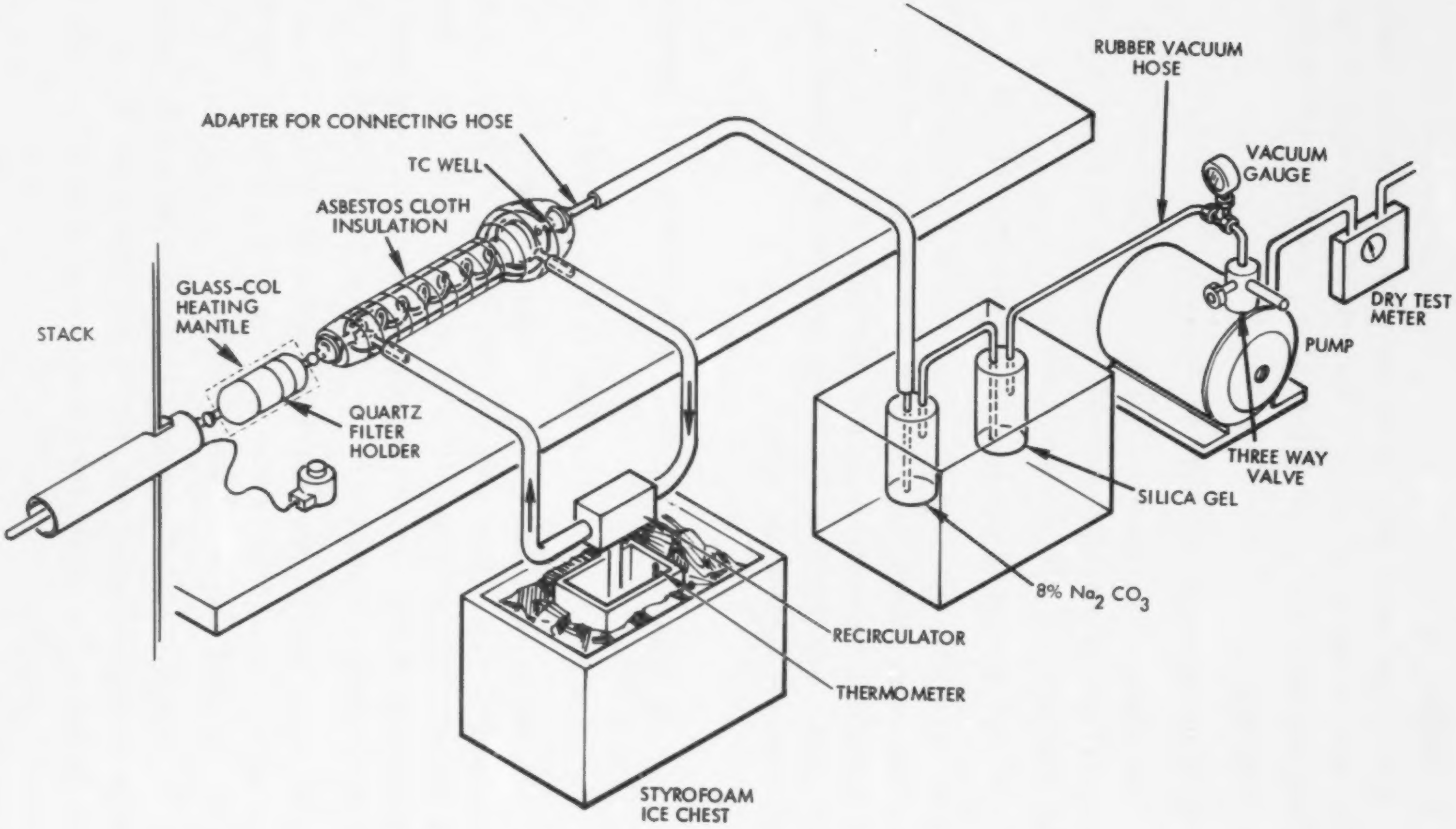

ADAPTER FOR CONNECTING HOSE
TC WELL
ASBESTOS CLOTH INSULATION
GLASS-COL HEATING MANTLE
STACK
QUARTZ FILTER HOLDER
RUBBER VACUUM HOSE
VACUUM GAUGE
DRY TEST METER
PUMP
THREE WAY VALVE
SILICA GEL
8% Na_2CO_3
RECIRCULATOR
THERMOMETER
STYROFOAM ICE CHEST

Figure 19. Controlled Condensation System Setup

3.3.6 Safety

OSHA safety requirements with regard to working environment and operator safety must be met at all times. The reagents mentioned in the procedure are not extremely toxic, but misuse of any chemicals can be harmful.

3.4 PROCEDURE

3.4.1 Probe Manufacture (Refer to Figure 20)

The necessary equipment is listed in Section 3.2.1.(a-m). Follow correct electrical safety procedures at all times. Be sure that no sharp pieces of metal abraid any of the electrical wires.

a) Cut the 304 SS one-inch tubing into 32-inch lengths.

b) Align the shielded thermocouple (TC) as shown in Figure 20. Using the glass tape, secure the shielded thermocouple to the Vycor probe. Place the unshielded thermocouple in the thermocouple well and secure with the glass tape. Continue down the probe, securing both thermocouple leads simultaneously against the tube.

NOTE

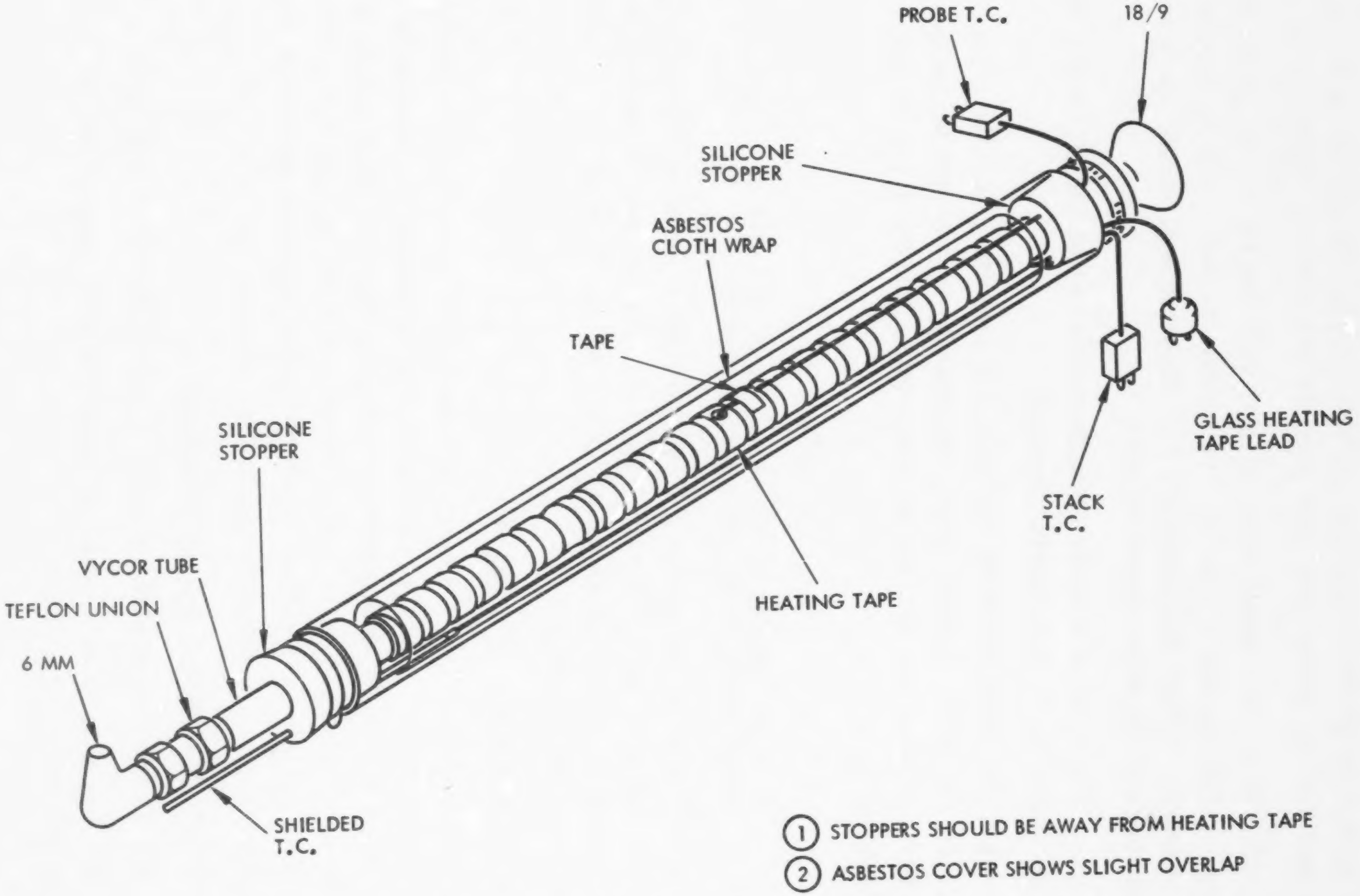
> Be careful never to kink the thermocouple or thermocouple leads.

c) Take the 72 inch glass heating tape and fold it in half.

d) Beginning 5 inches from the probe tip, wrap the probe with the glass heating tape. Make sure the heating tape is snugly up to the probe and secured every 6 inches with a wrapping of glass tape. Wrap the coils close enough so that the heating wire is completely used up 2 inches from the ball joint. Secure the end of the heating tape with a final wrap.

e) Bore a 9/16-inch hole into two No. 6 silicone rubber stoppers, then cut a slit vertically down one side of the stopper into the 9/16 inch hole. The slit will allow easy assembly.

f) Cut a piece of asbestos cloth approximately 30 inches long and wide enough to wrap the probe and heating tape with a 1/2 turn overlap. Tightly wrap the probe and secure the asbestos cloth with glass tape.


PROBE T.C.
18/9
SILICONE STOPPER
ASBESTOS CLOTH WRAP
TAPE
SILICONE STOPPER
GLASS HEATING TAPE LEAD
STACK T.C.
VYCOR TUBE
TEFLON UNION
6 MM
HEATING TAPE
SHIELDED T.C.

① STOPPERS SHOULD BE AWAY FROM HEATING TAPE

② ASBESTOS COVER SHOWS SLIGHT OVERLAP

Figure 20. Controlled Condensation System Probe Design

g) Slide the 304 SS sheath over the Vycor probe. Avoid scratching the insulation on the electrical leads. Position the sheath so that the end near the tip extends one inch past the start of the heating tape.

h) Spread the stopper open, slip it over the tip of the probe, and slide it into the 304 SS sheath. The stopper is then wired to help hold it in place. Repeat this procedure for the other end, except use a hose clamp to hold the back stopper in place.

i) Place the male quick connects on the end of the TC leads. The red TC lead goes to the negative terminal.

j) The probe should be tested in the laboratory to ensure that all parts are in order. Simply connect the heating wire to the Variac and allow the probe to heat up. Monitor the temperature to verify the TCs are functioning.

NOTE

Whenever heating up the probe, start off with very low power inputs (~5%) until heating starts.

k) The 0.25 inch nozzle and Teflon union (Figure 20) are attached prior to the test run. The nozzle consists of a 0.5 inch diameter quartz tube tapered to 0.25 inch at one end and a 90° bend placed in the center of its 2.5 inch length.

3.4.2 Filter Holder Fabrication

Figure 21 details the recommended design for the quartz filter holder. This filter holder consists of a modified 40/50 standard taper quartz joint. The modifications included adding a coarse quartz frit and an extension tube to the male joint to act as a pressure seal when the Tissue quartz filter pad is in place. Ball and socket (18/9) joints are used to connect the filter holder to the probe and controlled condensation coil.

3.4.3 Site Equipment Setup and Operation

a) In the 3-inch port, insert a 3 inch plug with 1 inch hole.

b) Use a table or another suitable device to support the CCS (see Figure 19).

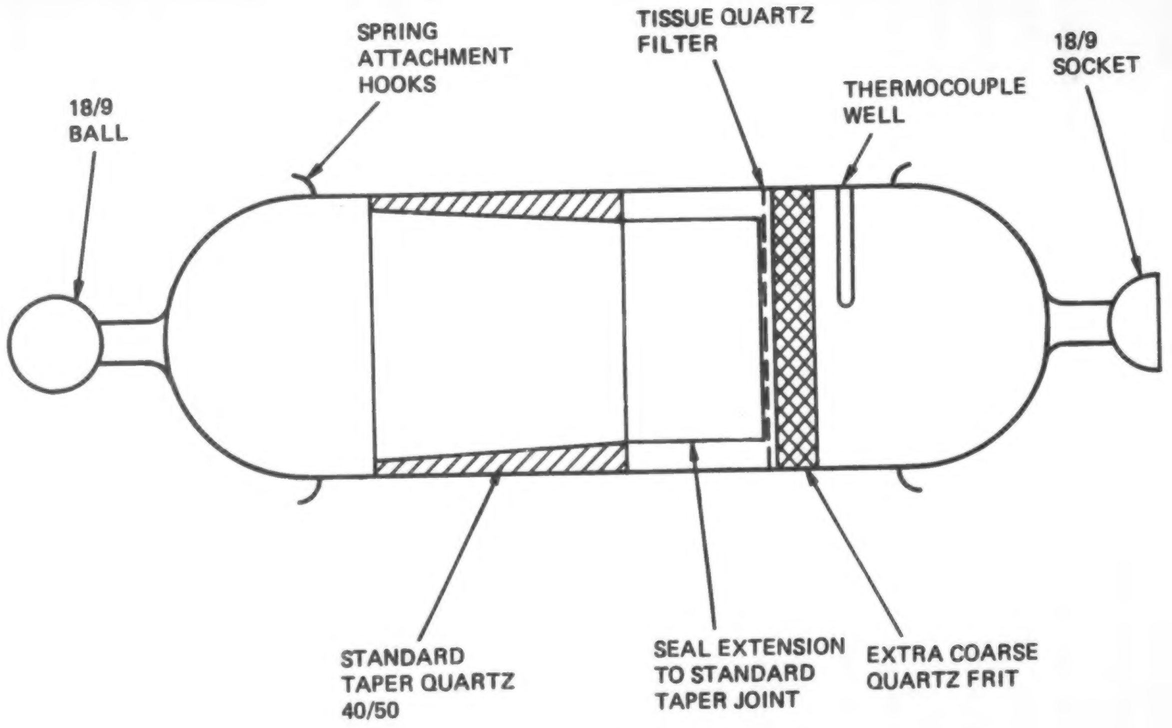

SPRING
ATTACHMENT
HOOKS
TISSUE QUARTZ
FILTER
18/9
SOCKET
18/9
BALL
THERMOCOUPLE
WELL
STANDARD
TAPER QUARTZ
40/50
SEAL EXTENSION
TO STANDARD
TAPER JOINT
EXTRA COARSE
QUARTZ FRIT

Figure 21. Quartz Filter Holder

c) Prior to use, be sure the controlled condensation coil (CCC) is clean and dry. Carry the CCC to the site with each end stoppered. If any condensation appears because of temperature changes, connect the CCC to the water bath and start the circulation of the 60°C (140°F) water. This should evaporate any premature condensate.

d) With the probe still out of the stack, assemble the train as shown in Figure 19. Be sure that each ball joint is completely clean and free of dust. Because of the possibility that the greases will freeze at the temperatures employed, it is not recommended that any grease be used. Proper care of the ground glass fittings will ensure that vacuum seals are maintained. Should any ground glass fitting not seal vacuum tight, a small amount of Apiezon H grease may be used for emergency repair. As soon as it is possible, the joint in question should be returned to the glass shop for regrinding (see Tables 7 and 8 for further suggestions).

e) Connect the soap bubble flowmeter to the vacuum pump exit. Be sure that the bubble flowmeter is vertical. Close off the end of probe with a stopper and turn on the vacuum pump and adjust the vacuum to read 380 torr (15 in. Hg).

f) Begin measuring the flowrate with the bubble gauge. If the leak rate is less than 85 mL/min (0.003 cfm), then the system is ready for use. If a leak rate greater than 85 mL/min is found, the system should be checked for loose joints and connections. The pump should also be checked and any worn parts replaced. Tables 7 and 8 for further information.

g) Once the vacuum test is completed, slowly turn the three-way valve to the vent position and allow the air to bleed into the system. This must be done carefully to prevent a pressure surge from backing up the impingers. Remove the bubble flowmeters from the system and unstopper the probe.

h) Begin heating the probe and the filter holder to 316°C (600°F) and 288°C (550°F) respectively. The heating bath should already be at 60°C (140°F). Once the skin temperatures reach these values, the run can commence.

NOTE

During the course of the run, the filter temperature will be controlled by the gas out temperature which should be 288°C (550°F).

i) After leak testing, the pump is again turned on and the flow rate adjusted to 10 Lpm (0.35 cfm). The pump is turned off without readjusting the valve settings.

j) Pinch the hose at the end of the controlled condensation coil and insert the heated probe into the duct with the nozzle pointed upstream.

k) Turn on the pump, release the pinched hose, and obtain an initial dry gas meter reading. Throughout the run, collect the data required (see Figure 22).

l) Sample for one hour or until 1/2 to 2/3 of the length of the coils are frosted with H_2SO_4.

NOTE

> If the coil is operating properly the H_2SO_4 will cover the inside of the coils as a thin gray-white film. If large drops of a clear liquid form and begin to block the coil, then moisture is being condensed. Either the percentage moisture has exceeded 16% or the temperature of the water bath has dropped below 60°C. Abort the run and check the water bath temperature with a Hg thermometer and confirm the percentage moisture in the gas stream. If the water bath is below 60°C, recalibrate the temperature bath control. For every percent above 16% H_2O, adjust the CCC temperature 2°C upward. Clean and dry the CCC, and replace the reagents in the impingers prior to restarting the run.

m) At the end of the sampling period remove the probe from the duct and slowly shut off the pump. After the pressure drops, remove the CCC from the system without removing the water bath hoses. Carefully connect (Figure 23) the G/R coil to the Erlenmeyer flask without spilling any condensate in the tube. In 10 mL increments (up to 30 mL), use deionized water to rinse out the CCC. Be careful to avoid introducing any dust or grease into the rinse solution. Take the rinse solution in the stoppered Erlenmeyer to the laboratory for analysis.

NOTE

> Multiple rinses are recommended to ensure a quantitative rinse of the coil.

AEROSOL SO_3
(CONTROLLED CONDENSATION)
FIELD DATA SHEET

Sample Location ______________

Run # ______________

Run Date/Time ______________

Operator ______________

Flowrate (cfm) ______________

Ambient Pressure (P) ______________

Reheater Air Flow Rate, acfm ______________

Inlet Gas Rate, acfm ______________

Sample Location SO_2 (ppm) ______________

Boiler Load (mw) ______________

Leak Rate ______________

Time (Min)	Temperature (°F)								Gas Meter Reading, cu. ft.
	Stack	Probe	Filter		Recirc. Water	Exit Coil	Dry Gas Meter		
			Skin	Out			In	Out	
AVG.									

Figure 22. Controlled Condensation Field Data Sheet

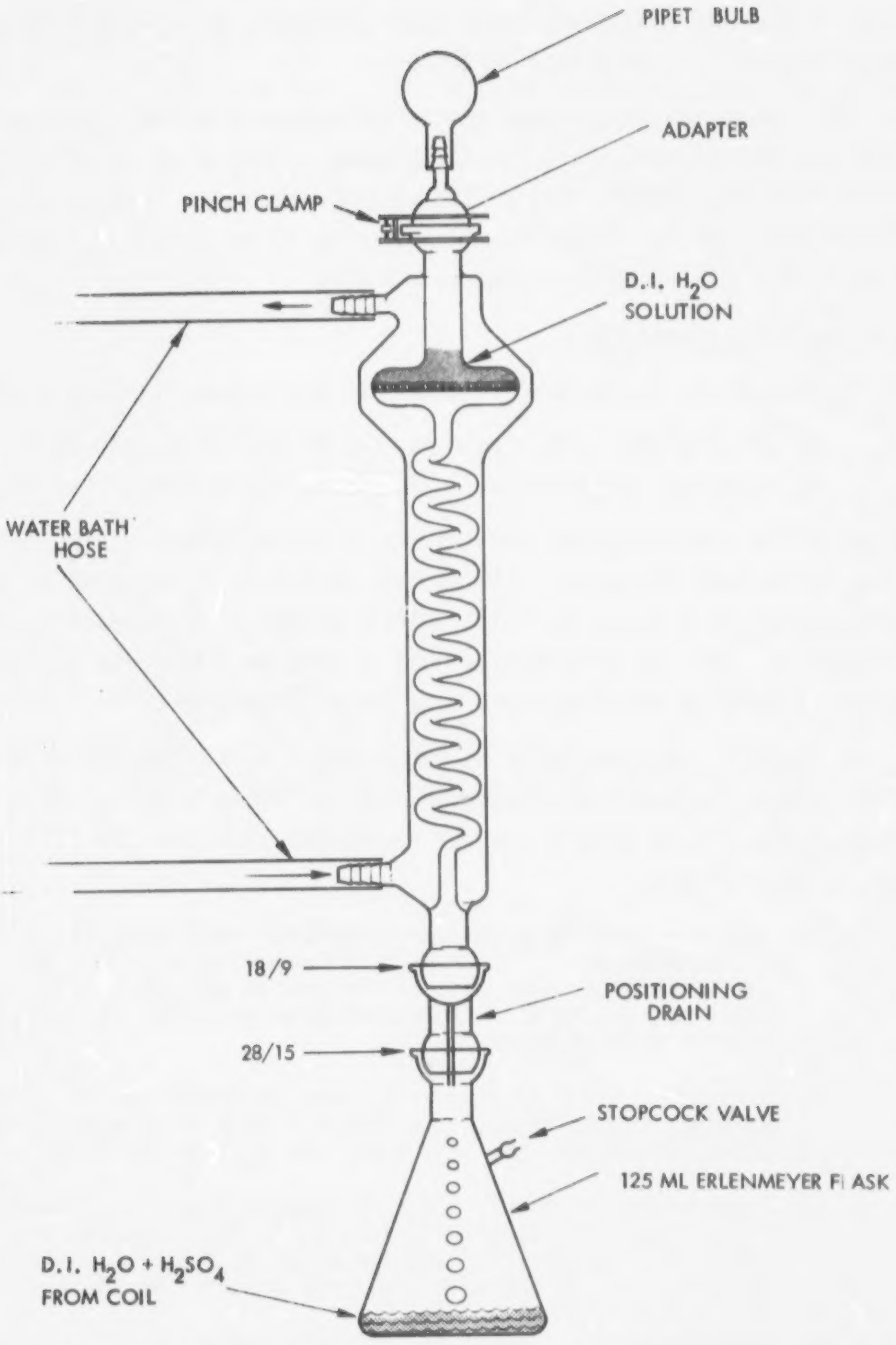

PIPET BULB
ADAPTER
PINCH CLAMP
D.I. H_2O SOLUTION
WATER BATH HOSE
18/9
POSITIONING DRAIN
28/15
STOPCOCK VALVE
125 ML ERLENMEYER FLASK
D.I. $H_2O + H_2SO_4$ FROM COIL

Figure 23. Controlled Condensation Coil Rinsing Apparatus

n) Rinse the probe with 30-40 mL of deionized H_2O after it has cooled. Take this solution back to the laboratory, and filter through a Whatman number 1 filter dilute to 50 mL.

o) Remove the filter from the filter holder (CAUTION: Wait until the filter has cooled), and place it into a beaker. Add 30 mL of deionized H_2O and swirl the beaker. Filter the solution through a Whatman number 1 filter into a 50 mL volumetric. Repeat with 10 mL portions of deionized H_2O until the volumetric is filled to the mark.

3.4.4 Analysis Procedures

Two procedures can be used to determine the amount of H_2SO_4 collected:

1) An acid/base titration using Bromophenol blue indicator or
2) A sulfate titration using Sulfonazo III as the indicator.

Because of the simplicity and sensitivity of the acid/base titration, it is the recommended procedure. The sulfate procedure is included in this section to act as a backup or total sulfate method if the need arises. In either case all the titrations should be done in triplicate and the results recorded on the laboratory data sheet (Figure 24).

a) Sulfate Titration Using Sulfonazo III. This procedure is similar to the sulfate procedure developed for scrubber liquors (Ref. 3.6). This procedure may also be used to analyze the water rinse from the filter for water soluble sulfate.

1) Wash the Dowex 50 W-X8 cation exchange resin with 10% V/V HCl. Fill a 1/2-inch I.D. ion exchange column to a 3-inch bed depth, and place glass wool pads at the bottom and top of the bed. Rinse the column with deionized water until the eluant tests neutral with pH paper.

2) Transfer 0.025 g of chemically pure Sulfonazo III indicator $[(NaSO_2)_2 C_{10} H_2(OH)_2](N{:}NC_6 H_4SO_3H)_2$ to a 25 mL bottle, add water to dissolve the indicator, and fill to the mark.

3) Transfer approximately 3.9 g of reagent grade barium perchlorate trihydrate $[Ba(C\ell O_4)_2 \cdot 3H_2O]$ into a one liter reagent bottle, add a small amount of distilled water to dissolve the salts, and then fill to the mark. Mix the contents of the bottle.

4) Standardize the reagent by titrating sodium sulfate. Dry the Na_2SO_4 in an oven for two hours at 125°C and allow to cool to room temperature in a desiccator. Weigh out accurately in

AEROSOL SO_3
(CONTROLLED CONDENSATION)
LABORATORY DATA SHEET

Run #________________________ Sample Location________________________

Run Date/Time________________________ Date Lab Analysis Completed ______________

Analyst________________________

Variable	Value
Aliquot Size (A)	________________ (mL)
Normality of titrant (N)	________________ (eq/ℓ)
ml of titrant used to titrate G/R coil rinses (v)	________________ (mL)
	________________ (mL)
	________________ (mL)
	Avg. ________________ (mL)
Blank (equivalent NaOH)	________________ (mL)
Net titration volume (V)	________________ (mL)
Absolute dry gas meter temperature (T_m)	________________ (^oR)
Volume of gas sampled (V_m)	________________ (ft^3)
Meter Pressure (P_m)	________________ (in. Hg)
ppm H_2SO_4 (vol/vol)	________________

Normality of acid used to titrate blank (if used) ________________

$$\text{ppm } H_2SO_4 = 1202.52 \times \frac{NVT_m}{AV_mP}$$

Figure 24. Laboratory Data Sheet for Acid/Base Titration

triplicate 12 to 16 mg of the Na_2SO_4 from a weighing bottle into 125 ml Erlenmeyer flasks, dissolve with 10 mL deionized water, add 10 mL acetone and three drops Sulfonazo III indicator solution, and titrate with the barium perchlorate.

5) Repeat this procedure in triplicate for the sample and blank D.I., (deionized H_2O):

$$M = \frac{W}{(142)\ (V - v_a)} \qquad (3\text{-}1)$$

Where: M = Molarity of the barium perchlorate solution, moles/liter

W = Weight of sodium sulfate titrated, mg

V = Average volume of barium perchlorate solution required for titration of sodium sulfate, mL

v_a = Average volume of deionized water blank titration

6) Take a 10 mL aliquot of the rinse solution and pass it through the ion exchange column at 3 mL/min. Rinse the column with 30 mL deionized water and collect the eluant and rinsings in a 50 mL volumetric flask and dilute to the mark with deionized water.

7) After every tenth use of the column, regenerate it with 100 mL of 10% W/V HCl at 3 mL/min flow rate and rinse until the eluant tests neutral to pH paper. Rinse the column with 50 mL of deionized water.

8) Add 10 mL acetone and three drops of the Sulfonazo III indicator to a 10 mL aliquot of the ion exchange eluant.

9) Titrate with 0.01 M $Ba(ClO_4)_2$ using a magnetic stirrer and back lighting. The color will change from purple to blue. The end point is the point at which an additional drop of titrant does not change the color of the solution. The end point should not fade unless left standing for more than 5-10 minutes. Record the volume of 0.01 M $Ba(ClO_4)_2$ used to reach the end point and calculate the average titration volume. Titrate a 10 ml aliquot of deionized water in the same fashion to obtain the titration blank.

b) Acid/Base Titration. The preferred method of analysis is the acid/base titration using Bromophenol blue indicator. Carefully handle and store the samples in clean glassware and analyze them as soon as possible. Record all results on the laboratory data sheet.

1) Pipet 10 mL of the 0.0200 N KHP solution into a 125 mL wide-mouth Erlenmeyer Flask.

2) Add 3 drops of the phenolphthalein indicator. With a swirling action of the flask, titrate with 0.02 N NaOH solution until the first pink color stays. Record the volume and repeat from (1) in triplicate. Repeat this procedure using deionized H_2O (blank).

3) Average the colume used to titrate the KHP solution. The true normality of the NaOH solution equals:

$$N = \frac{(0.200)\ (10\ \text{mL})}{(\text{mL titrant-mL blank})} \qquad (3\text{-}2)$$

4) To titrate the H_2SO_4 in the condensation coil, probe and filter rinses, pipet 10 mL of one of these solutions into a 125 mL Erlenmeyer flask. Larger aliquots can be used if the H_2SO_4 is quite low. As a rule of thumb, the aliquot size should be adjusted to require a minimum of 1 mL of titrant.

NOTE

Be sure to use the same size aliquot for the blank titration.

c) Calculation of the ppm (v/v) concentration of H_2SO_4 in the Gas streams. Using either the sulfate or acid/base titration, to analyze the CCC rinse, the concentration of H_2SO_4 can be calculated.

1) From the Field Data Sheet (Figure 22) obtain the average dry test meter temperature, volume of gas sampled and atmospheric pressure. Record these values on the Laboratory Data Sheet.

2) Using the Laboratory Data Sheet, insert the correct numbers into the appropriate formula (see Appendix B for the derivation):

For the Acid/Base Titration:

$$\text{ppm } H_2SO_4 = 1{,}201.9\left(\frac{NVT_m}{AV_mP_m}\right) \qquad (3\text{-}3)$$

For the Sulfate Titration:

$$\text{ppm } H_2SO_4 = 12{,}019\left(\frac{MVT_m}{AV_mP_m}\right) \qquad (3\text{-}4)$$

The result is ppm (v/v) H_2SO_4 at 20°C (68°F) and 1 atm (29.92 in Hg).

3) Plot this data on the CCS control chart (see Section 4.2.3-b) and record the results on the Laboratory Data Sheet.

4. QUALITY ASSURANCE METHODOLOGY

The intensive test program being carried out at the Shawnee Test Facility is designed to determine whether scrubber conditions affect the quantity and quality (as measured by a particle size) of emitted particulates. In addition, a concurrent program of monitoring H_2SO_4 has been initiated.

To accomplish these tasks, a series of procedures for particle sizing and H_2SO_4 measurement were written. This quality control document is written as a supplement to those procedures to provide guidance to on-site personnel in controlling the quality of their work.

This document is not designed to be a procedure manual and consequently does not contain detailed information on the procedure. What is provided is information on:

- Equipment care and usage
- Guidelines for laboratory techniques
- Specific critical area checklists for each procedure
- Data interpretation aids to monitor the quality of the results achieved
- Specific maintenance and calibration schedules
- Troubleshooting and repair schemes.

No matter how good a document is, the degree of implementation will establish its usefulness. If the spirit of the document is violated, then the quality of the results will not be improved. Committing a mistake during the operation of the test equipment is regrettable, but not documenting the problem is unforgivable. The purpose of this document is not to assign blame, but to provide a basis for understanding and interpreting program results. Always remember that quality control ultimately rests with the honesty of the operating personnel, and no piece of paper can replace a dedicated professional.

4.1 LABORATORY EQUIPMENT CARE AND TECHNIQUE

To assure the quality and reliability of all data generated in this program, it is of utmost importance that all equipment is in proper working order. This encompasses not only routine periodic maintenance but also the day-to-day handling and general usage of this equipment in a safe and secure manner. The methodologies described in each of the following categories are aimed to help the operator maintain his equipment in an adequate manner so that it is always ready to operate with a certain measure of reliability.

The laboratory techniques to be used will have a direct relationship to the type of end-data which are obtained. To generate better, more reliable data, certain techniques should be used throughout this program. The specific techniques are tabulated as they relate to the particular apparatus or glassware employed in the performance of that test.

Cleanliness is one of the major factors affecting the quality and accuracy of data. It is of utmost importance since cross-contamination will be minimized just by having glassware and equipment available in a clean, non-contaminated condition. At the minimum:

- An area should be wiped down prior to use of that area.
- A Whatman No. 1 paper sheet (46 x 47 cm) shall be placed on the bench top prior to working in that area.

4.1.1 Analytical Balance

a) Equipment Care

Analytical balances are a relatively fragile type of instrument, and are subject to shock, temperature and humidity changes, general mishandling and various other potentially injurious occurrences. Some of the precautions to be observed in maintaining and prolonging the dependable life of a balance are as follows:

- Analytical balances should be mounted on a heavy shock proof table, preferably one with adequate working surface and a suitable drawer for storage of balance accessories.
- Balance level should be checked frequently and adjusted when necessary.

- Balances should be located away from laboratory traffic, protected from sudden drafts and humidity changes.

- Balance temperatures should be equilibrated with room temperature; this is especially important if building heat is shut off or reduced during non-working hours.

- When not in use, the beam should be raised from the knife edges, all dials set to zero, objects such as weighing dish removed from the pan, and the sliding door closed.

- Never add any weights or samples to the pan unless the beam is raised from the knife edges (half release position)

- Place a petri dish with desiccant in the balance pan area.

- Special precautions should be taken to avoid spillage of corrosive chemicals on the pan or inside the balance case. The interior of balance housing should be kept scrupulously clean; a camel hair brush should be used to clean the balance pan.

- Balances should be checked and adjusted periodically by a company service man or balance consultant. If service is not available locally, follow the manufacturer's instructions as closely as possible.

- The balance should be operated at all times according to the manufacturer's instructions, (which are to be posted on or near the balance).

b) Laboratory Technique

Since the analytical balance is a very sensitive instrument, special precautions or specialized techniques must be followed:

- Only weigh samples which are at room temperature.

- Never touch weights, pan, samples, etc., with your hands as they would deposit a thin layer of oil and cause resultant errors.

- Verify the balance is clean and no contaminants are on the pan. In the event some contaminant, dust, dirt, sample, etc., is on the pan, remove that contaminant by brushing it off. If oil or water is on the pan, wipe it off using an acetone soaked tissue.

- Level and zero the analytical balance prior to weighing.

- Never put chemicals directly on balance pan.

- The same person should pre-weigh, manipulate and reweigh a given set of filters and liners from an impactor run.
- Weigh each sample as quickly as possible (within the constraint of good technique) to avoid weight changes due to moisture pickup.
- Always keep the balance doors closed while weighing.
- Always add or subtract weights while the beam is raised from the knife edges.
- Record all weights immediately.

4.1.2 pH Meters

a) Equipment Care

A basic pH meter consists of a voltage source, amplifier, and readout device, either scale or digital. Certain additional refinements produce varying performance characteristics between models. Some models incorporate expanded scales for increased readability and solid state circuitry for operating stability and extreme accuracy. All instruments of recent design also include temperature adjustment and slope adjustment to correct for asymmetric potential of glass electrodes. Other features are scales that facilitate use of selective ion electrodes, recorder output, and interfacing with complex data handling systems.

- The pH meter should be kept on top of a counter in a work area.
- Glass electrodes should not be allowed to become dry during periods of inactivity. When not in use they should be immersed in distilled water, with the water level being checked frequently.
- The proper KCl level in the calomel electrode shall be maintained.
- Prevent contact of the electrodes with oily substances or other type of materials which could adhere to the electrode surfaces. In event contact is made, clean the electrode with acetone followed by deionized water. Allow the electrode to equilibrate in a pH 4 buffer until it is stable.

b) Laboratory Technique

pH meters are highly dependent on calibration and general use techniques. Essentially, the major area of concern is the proper use of the electrodes and reference pH solutions. Following are the various techniques which should be adhered to in order to assure good standardization and pH readings.

- The first step in standardization of the instrument is done by immersing the glass and calomel electrodes into a buffer of known pH, setting the meter scale or needle to the pH of the buffer and adjusting the proper controls to bring the circuit into balance. The temperature compensating dial should be set at the standard solution temperature. The pH of the standard buffer should be within about two pH units of the sample.
- The instrument should be calibrated against two buffers that bracket the pH of the samples. If the two standards do not read accurate values, a troubleshooting mode of operation should take place to ascertain why not.
- A new slope setting must be made whenever electrodes are either changed, subjected to vigorous cleaning, or refilled with fresh electrolyte.
- Glass electrodes have a very fast response time in highly buffered solutions. However, accurate readings are obtained slowly in poorly buffered samples, and particularly when changing from buffered to unbuffered samples, as after standardization. Electrodes, both glass and calomel, should be well rinsed with distilled water after each reading, and should be rinsed or dipped several times into the next test sample before the final reading is taken. Weakly buffered samples should be stirred during measurement.

4.1.3 Laboratory Analytical Glassware

a) Equipment Care

All glassware is somewhat fragile and requires good handling, storage and inspection criteria.

- Try to avoid bumping glassware or otherwise causing it to become stressed, cracked, or broken.
- Keep all glassware on shelves with special lips or in drawers; this will prevent glassware from rolling off the counter.

- Any cracked or broken glassware should be discarded and replaced at the direction of the supervisor.
- Never place dirty glassware near the cleaned glassware.
- Never store dirty glassware. Rinse all glassware with acetone and water, cleaning thoroughly the next day.
- Volumetric flasks should not be dried in an oven, but rather final rinsed in deionized water and stored with some deionized water in it.
- Grease any vacuum joints using a minimum amount of grease.

b) Laboratory Technique

- Always rinse glassware with acetone to remove any organic substances.
- Rinse with tap water.
- Clean all glassware with either Alconox or Calgon in water. Use a brush to scrub all surfaces.
- After either the soap wash or chromic acid treatment, throughly rinse the glassware with abundant amounts of tap water.
- Several separate rinses will be required to remove all soap and traces of the chromic acid.
- Rinse the glassware with deionized water a minimum of three times. Water should sheet off. Otherwise the glassware is not clean and the entire process must be repeated.
- Air dry the glassware. CAUTION - do not blow dry the glassware with lab air as that air will put an oily film back onto the glassware.

c) Glassware Usage

Use of glassware essentially revolves around volumetric glassware or any glassware involved in critical measurements. Guidelines follow which will ensure better precision and accuracy.

- The volumetric apparatus must be read correctly. The bottom of the meniscus should be tangent to the calibration mark.
- To deliver (TD) volumetric pipets are calibrated to deliver a fixed volume. In emptying volumetric pipets, they should be held in a vertical position and the outflow should be

unrestricted. The tip of the pipet is kept in contact with the wall of the receiving vessel for a second or two after the free flow has stopped. This will remove any hanging drops. However, do not blow out any remaining solution from the tip of the pipet. Do not attempt to dry a pipet which has been used, simply rinse the pipet with the new solution to be used several times; increase the number of rinses if going from a concentrated to a dilute solution.

- Burets are used to deliver definite volumes. General rules in regard to the manipulation of a buret are as follows: a) Do not attempt to dry a buret which has been cleaned for use, but rinse it two or three times with a small volume of the solution with which it is to be filled. b) Do not allow alkaline solutions to stand in a buret, because the glass will be attacked, and the stopcock, unless made of Teflon, will tend to freeze. c) Burets should not be emptied rapidly. Otherwise too much liquid will adhere to the walls and as the solution drains down, the meniscus will gradually rise, giving a high false reading. d) It should be emphasized that improper use of and/or reading of burets can result in serious calculation errors.

- In the case of all apparatus for delivering liquids, the glass must be absolutely clean so that the liquid film never breaks at any point. Careful attention must be paid to this fact or the required amount of solution will not be delivered.

4.1.4 Desiccators

a) Equipment Care

Desiccators are glassware with a specific use. They should be treated as glass apparatus with the following added requirements:

- Maintain the desiccator in a clean condition. Periodic cleaning will necessitate removing Drierite, thoroughly cleaning the unit, then drying it in an oven at 110°C for a minimum of two hours and putting in fresh desiccant.

- Use Drierite or silica gel, and it should be placed at the bottom of the desiccator under the porcelain plate.

- Never allow any desiccant to be placed on top of the porcelain plate.

- Add an indicating Drierite or silica gel to monitor the effectiveness of the desiccant. If the indicator is blue, usage may continue; if indicator is pink, the desiccant must be replaced or regenerated.

- Always keep a lid on top of desiccator.
- Maintain seal between lid and base of desiccator by placing a small amount of Dow 111 High Vacuum silicone grease on the mating surface and rotating the lid on the base 360^0.

b) Laboratory Technique

Desiccators are used to store samples or glassware whenever moisture must be eliminated.

- Adhere to precautions of desiccator in "equipment care" section.
- Do not allow samples to touch the grease on the mating surface of the lid and base of desiccator.
- Open lid of desiccator by sliding lid sideways until it is off. Never try to pry open or lift straight up.
- Keep lid on desiccator except for minimal time needed to transfer samples in and out of desiccator.
- Do not put solvents into dessicator.
- Always let samples cool to room temperature inside the dessicator before removing them for weighing or performing other temperature and moisture sensitive tests.

4.1.5 Dry Test Meters

a) Equipment Care

Drv test meters are relatively insensitive to handling; however, several precautions should be noted and adhered to.

- The meter should not be dropped or handled roughly.
- Always maintain the meter in a horizontal position while flow measurements are made.
- The unit must be periodically calibrated for proper gas flow readings (see maintenance schedule for recommended schedule).
- The meter should always be placed downstream of the impingers and drying train to prevent acid gases and moisture from reaching the dry test meter.

4.1.6 Ovens

a) Equipment Care

- Ovens should always be kept clean. Any spills or other contamination shall result in a thorough cleaning with all items being removed from the oven.
- High outgassing products or solvents should not be placed in the oven.
- Dirty glassware or hardware shall never be placed inside an oven.
- Never place tubing or other plastics inside an oven.
- Periodic checks on the oven will be required to verify its temperature readout control is in calibration and that a uniform temperature exists inside the oven. This task should be done monthly.

4.1.7 Reagent Storage

It is very important that all reagents, solvents and standard solutions be stored in an appropriate manner to prevent contamination and/or deterioration of that material prior to their use. All reagents should be clearly labeled as to the material and concentration as well as the date standardized and the performing technician.

- Borosilicate glass bottles with ground glass stoppers are recommended for most standard solutions and solvents.
- Plastic containers, e.g., polyethylene, are recommended for alkaline solutions. Plastic containers must not be used for reagents or solvents intended for organic analyses.
- It is important that all containers be properly cleaned and stored prior to use. (See 4.1.1)
- Standard reagents, solvents, and other chemicals must always be stored according to the manufacturer's directions. Reagents or solvents that are sensitive to the light should be stored in dark bottles and/or stored in a cool, dark place.
- The analyst should pay particular attention to the stability of the standard reagents. Reagents should not be kept longer than recommended by the manufacturer or as normally used in the method selected.

- The concentration of the standards will change as a result of evaporation of solvent. This is especially true of standards prepared in volatile organic solvents. Therefore, the reagent bottles should be kept stoppered, except when actually in use.

- Storage should conform to OSHA safety practices.

4.1.8 Blanks

Two types of blanks exist:

- Individual blanks - the blank of each chemical such as acetone used to flush the samples from the impactors, the deionized water and any and all other solvents used.

- Method blank - the method blank is determined by following the procedure step-by-step, including all of the reagents and solvents, in the quantity required by the method.

a) Laboratory Technique

In general these guidelines should be followed to monitor blanks:

- Blanks should be run on each different individual type of sample and on each batch of samples.

- The conditions for determining the blank must be identical to those used throughout the analysis, including the detection system.

- If any individual blanks are found to interfere with the analysis, the cause of interference will have to either be determined or a correction factor applied (if it is found that a bias results).

- If the cumulative blank interferes with the determination, steps must be taken to eliminate or reduce the interference to a level that will permit this combination of solvents and reagents to be used. If the blank cannot be eliminated, the magnitude of the interference must be considered when calculating the concentration of specific constituents in the samples being analyzed. Within the program at Shawnee there are two examples of blanks.

    1) Weighing blanks (see below)
    2) Indicator blanks (see Section 4.1.9-b)

Proper implementation of blanks in these two areas will ensure a successful analysis.

b) Weighing Blanks

- Always weigh a fresh set of blank impactor plates and filters which have been greased and otherwise subjected to the same conditions as field samples, except that they are not used to collect samples.
- Obtain a known standard weight (about 1 g) and weigh that standard at least once a day while weighing actual specimens. Maintain this sample in the desiccator between weighings. Record this weight and maintain it in a separate log book. This weight should be plotted to ensure that only random changes are occurring. If positive or negative trends occur, review the weighing procedures and/or call in the balance service man for a calibration check.
- If a sample blank varies by more than 0.0001 g, then a correction must be made on the sample weights. Weight gains should be subtracted from all the samples in the blank's group while any weight loss in the blank should be added to the actual sample.

NOTE

Always inspect the weighing blanks for any obvious sign of contamination, such as dirt particles or the loss either of grease or glass fibers due to handling. If an obvious contamination is noted, do not correct the samples, but note the reason for the change in weight of the blank.

4.1.9 Titrations

a) Laboratory Technique

End points for titrations are very color dependent, i.e., the end point will probably vary slightly for each operator's sense of color. To obtain the most accurate data, the following techniques should be employed in all titrations:

- Always add the same number of drops of indicator.
- Have the same operator do blank and sample.
- Always titrate to the same color intensity.
- Avoid parallax errors - keep eyes at the same level as the liquid meniscus and hold a white piece of paper behind it with a dark line horizontal to the table top.

- Remove air bubbles from buret tip prior to use.
- Never store reagent in buret. Always rinse out buret with a slight amount of titrant.
- Always record titrant type and volume used.

b) Indicator Blanks

Each indicator will change color over a different pH range. For example:

Indicator	pH range	Color change
Bromophenol Blue	3.0-4.6	yellow to blue
Phenolphthalein	8.2-10.0	colorless to pink

The point measured by the indicator is simply the point at which the color change occurs. The actual end point where exactly the right amount of acid and base have reacted (equivalence point) can be close to or far away from the indicator end point. Thus Bromophenol blue is chosen for the NaOH + H_2SO_4 titration, since the equivalence point occurs at about pH 3. Phenolphthalien is used for the potassium hydrogen phthalate + NaOH standardization titration because the equivalence point is near pH 7.

Even though the indicators have been selected to be as close as possible to the actual end point, a small difference still exists and is called the indicator blank. The indicator blank for phenolphtalein is the amount of NaOH required to change a specific amount of water containing a known number of drops of phenolphtalien pink. This value is subtracted from the milliliters used to titrate the sample.

The indicator blank for Bromophenol blue is determined in the same way (known volume and number of drops) except that a standard acid (H_2SO_4) is used to backtitrate the indicator in distilled water to a yellow color. The number of milliequivalents used is added to the amount found titrating the sample.

NOTE

Blanks can vary with sample size and number of drops of indicator, therefore determine the indicator blank under the same conditions in which the sample is titrated.

4.1.10 Handling

a) Laboratory Technique

Handling techniques are very critical to the ultimate success of the program, mostly in terms of obtaining more reliable data, but sometimes even in terms of getting data.

- Care must be taken to limit contact with the impactor discs. At no time should the discs be touched with ungloved hands. All of the laboratory manipulations are to be done in a clean environment using tweezers to handle discs. Remember, several grains of dust could represent the total weight captured on a disc. Contamination control is essential during greasing, drying and weighing.
- Handling of glassware is very sensitive and care should be taken to avoid any shock, bumping or strain of the glassware.
- Do not touch grease or components with grease on them to other hardware.
- Always use gloves, but be careful that organic solvents do not come in contact with the gloves, otherwise the gloves might discolor. Do not use gloves that are powdered by the manufacturer.
- Mechanical shock to hardware should be avoided. This is especially important in the G/R system where high temperatures reduce the resistance to mechanical shock.

4.2 SAMPLING QUALITY CONTROL

An impactor operates under the principle that if a stream of particle-laden air is directed at a surface, particles of sufficient inertia will impact upon the surface while smaller particles will follow the air stream lines and not be collected. Thus an impactor consists simply of a nozzle and an impaction plate. Each stage of an impactor positions the nozzle a precise distance above the impactor plate. Each successive jet is smaller in diameter so that the gas velocity increases and smaller particles are collected. To minimize particles bouncing off of the surface of the collection plate, the impactor surface is coated with a sticky material. The presence of the sticky material also minimizes re-entrainment of collected particles by the scouring action of the gas stream. The best approach to reducing re-entrainment is not to overload the stages with collected material.

4.2.1 Brink Methodology

The impactor selected to measure the particle size distribution entering the wet scrubber system is the Brink impactor. The Brink impactor system consists of a 1/4-inch ID, 5-foot probe connected directly to the impactor (Figure 1). The impactor has been modified by the addition of a cyclone placed before the first stage. This cyclone is in addition to the five stages already present in the impactor. A final stage consisting of a 47 mm filter is attached to the exit of the last impaction stage. This Brink system will provide aerodynamic size information for particles from 0.3μ to 10μ. With Teflon washers and the Apiezon H greased stages, the maximum operation temperature is 200°C (392°F) at a maximum flow rate of 0.08 acfm.

The Brink system is an out-of-stack extractive sizing method. Using an Aerotherm probe, a velocity profile for the duct is obtained. The average velocity is calculated and used to select a nozzle that will sample at the average velocity isokinetically at a flow rate of <0.08 acfm.

Temperature control of the impactor and filter system is maintained by monitoring the inlet and outlet gas temperature from the impactor. The necessary heat is supplied by a specially designed Glass-Col heating mantle.

The gas flow rate is monitored by measuring the ΔP across the impactor with a magnehelic gauge. The impactor acts as a calibrated orifice and thus the ΔP can be related to flow rates by referring to calibration charts provided by the manufacturer.

The amount of material collected is determined by weighing the collection stages before and after the run. Probe and tubing rinses are added to the cyclone catch. The collection plates and filter are thermally conditioned and desiccated prior to weighing. After sampling, the samples are desiccated to constant moisture content prior to re-weighing. Because of the potential for random or systematic errors in weighing, blanks consisting of spare collection stages and filters are conditioned and weighed along with the samples to monitor weighing errors. Refer to Section 4.1.8 for the proper use of blanks.

a) Critical Checkpoints

Table 4 is a checklist of critical items that should be followed during the test run. These critical items consist of:

- Recommended flow rates, temperatures and sampling times
- Reminders on laboratory and sampling technique
- Specific equipment checks.

While this list is provided for review prior to the sampling run, its best use is an on-site checklist for the supervisor and quality assurance personnel during the run. During a test audit the supervisor or QA representative should initial each item successfully completed. The entire list should be included with the documentation of that test run. The operating personnel might also like to have copies of the checklist for reference during the execution of the test run. Copies can be posted in the laboratory and sampling site for this purpose.

b) Data Monitoring Procedures

Dry aerosol sampling procedures cannot be tested in the classical fashion, i.e., with spiked (standard addition) samples to determine their reliability. There are simple monitoring activities that can be carried out on a daily basis. These activities include:

- Calculation of percent isokinetic sampling - provides information on the quality of sampling,
- Comparison of Aerotherm and Brink grain loadings - determines the efficiency of particle recovery from system.
- Comparison of Aerotherm and Brink fine particle grain loadings - indicates the efficiency of sampling by the impactor.

These procedures are discussed in detail in the following paragraphs.

1. Isokinetic Sampling Tests

Isokinetic sampling is sampling at a rate (measured at the probe nozzle) equal to the velocity of the gas flowing by the probe. Unless gas flows are sampled isokinetically, larger or samller particles can be preferentially collected depending on whether the sample rate is less than or greater than the stream flow rate. The closer to isokinetic conditions the more representative the gas particle sample will be.

TABLE 4. CRITICAL CHECKPOINTS FOR BRINK DRY AEROSOL SYSTEM

Checkpoint	Initials		Remarks
	Supervisor	QA Inspector	
I. Conditioning and Preweighing Procedures:			
A. Conditioning			
- Plates cleaned with toluene prior to greasing			
- Hands gloved.			
- Thin film of grease applied to plate.			
- Clean filter handled with tweezers.			
- Clean petri dishes and watch plates prior to storing impactor plates, cyclone or filter.			
- Plates, cyclone and filter placed in clean, labeled petri dish and covered.			
- Plates and cyclone conditioned at 175°C for 4 hours.			
- Filter conditioned at 290°C for 4 hours.			
- Desiccant fresh (color B/P?)			
- Plates, cyclone and filter desiccated for 2 hours.			
B. Weighing			
- Balance area clean.			
- Balance pan clean.			
- Balance leveled.			
- Balance zeroed.			
- How long were petri dishes and impactor stages left open to lab air? (min?).			
- Weigh impactor plates and filter blanks for each set of sample impactor plates and filter.			
- Data recorded on correct data sheets.			
- Note condition of plates (color of grease, thickness of coating, etc.) on data sheet.			
II. Laboratory Impactor Preparation			
- Plate, cyclone and filter identifications and weights recorded.			
- Impactor inspected for wear.			
- Impactor cleaned.			
- Work area cleaned and bench covered with Whatman paper.			
- Hands gloved.			
- Impactor stages loaded, starting from last stage.			
- Cyclone cup in place.			
- Backing, filter, and seal washer placed in the filter holder.			
- Inlet tubing sealed off.			

-Continued-

TABLE 4. CRITICAL CHECKPOINTS FOR BRINK DRY AEROSOL SYSTEM (CONTINUED)

Checkpoint	Initials		Remarks
	Supervisor	QA Inspector	
III. Site Set-Up			
- Impactor maintained in vertical position during transfer to site.			
- Brush inside probe prior to run.			
- Rinse probe with acetone until rinse solution is clear.			
- Fresh solutions placed in impingers.			
- Fresh absorbant replaced in final impinger.			
- Leak rate must be less than 0.0008 cfm (0.02 Lpm).			
- Leak test performed.			
- Magnehelic gauges zeroed.			
- Thermocouple leads attached to impactor.			
- Skin temperature controlled to < 375°F (< 191°C).			
IV. Sampling Run			
- Brink gas out temperature maintained at highest stack reading +50°F.			
- Brink gas out temperature must never exceed 347°F (175°C).			
- Check seal between probe and rubber stopper to prevent any outside air entering the stack.			
- Select sampling rates below 0.08 cfm (2 Lpm).			
- Select sampling time to collect no more than 10 mg of material on any stage except the cyclone.			
- After probe is disconnected, plug the ends to prevent particle loss during transfer to lab.			
- Impactor carried in an upright position to laboratory.			
- Support equipment cleaned prior to next run.			
- Report any experimental problems or unusual occurrences on data sheet.			
V. Sample Recovery			
- Probe and impactor cooled to handling temperature.			
- Use gloves during removal procedure.			
- Probe and tubing connections inlet to the cyclone are brushed and rinsed with acetone until rinse stream is clean. Rinsings collected in Erlenmeyer flask and saved for weighing in tared 50 mL Erlenmeyer flask.			

-Continued-

TABLE 4. CRITICAL CHECKPOINTS FOR BRINK DRY AEROSOL SYSTEM (CONTINUED)

Checkpoint	Initials		Remarks
	Supervisor	QA Inspector	
- Use tweezers to remove impactor plates.			
- Inspect impactor walls and jet nozzles for particulate matter. Brush any particulate matter on the walls of jet nozzles onto the next stage.			
- Inspect filter holder for shreds of filter material.			
- Collect all pieces of filter material from filter holder and place with the intact filter for weighing.			
- Record all data on laboratory weighing sheet.			
- Correct sample weights for any change in the blank's weight.			
- Note any unusual operations.			
VI. Data Verification			
- Plot the daily percentage isokinetic for Brink runs (Y-axis for % isokinetic, X-axis day)			
- Plot Aerotherm and Brink grain loading values on a daily basis (Y-axis for grain loading, X-axis day).			
- Plot Aerotherm and Brink fine particle grain loading on a daily basis (Y-axis fine grain loading, X-axis day).			
- Plot sample blank weight change daily (Y-axis wt., X-axis day).			

Thus the degree of isokinetic sampling is an expression of the quality of the sampling run. The normal criteria of acceptability is ±10 percent of the current isokinetic flowrate.

Because the internal gas velocity determines the size cutoffs for the collection stages, the impactor must be run at one flow rate is only one size range of particles is to be deposited on a given stage. Consequently, the impactor is run at the average isokinetic flow rate.

The procedure for the isokinetic check follows:

a) From the original velocity profile (or actual velocity measured during the run), compute the average $\sqrt{\Delta P}$.

b) Calculate the average stack velocity:

$$\overline{V}_S = (85.48)\ C_p\ \overline{\sqrt{\Delta P}}\ \left[\left(\frac{\overline{T}_S+460}{P_S M_S}\right)\right]^{1/2} \qquad (4\text{-}1)$$

where

$\overline{V}_S$ = Average stack velocity (ft/sec)

C_p = Pitot tube coefficient

$\overline{\sqrt{\Delta P}}$ = Average square root of the velocity head (in. $H_2O)^{1/2}$

$\overline{T}_S$ = Average stack temperature (^oF)

P_S = Absolute stack gas pressure in. Hg)

M_S = Stack gas molecular wt (g/m) (29.5 inlet to wet scrubber)

c) Convert ΔP_c to ΔP_E

$$\Delta P_E = \Delta \overline{P}_c \left(\frac{M_a}{M_S}\right)\left(\frac{\overline{T}_I+460}{537}\right)\left(\frac{29.92}{P_{IA}}\right) \qquad (4\text{-}2)$$

where

ΔP_E = Brink pressure drop corrected for impactor conditions (in. Hg)

$\Delta \overline{P}_c$ = Average Brink pressure read during test run (in. Hg)

$\overline{T}_I$ = Average of the impactor gas in and out temperatures (^oF)

$\overline{P}_{IA}$ = Average pressure inlet to Brink impactor (in. Hg absolute)

M_a = Atmospheric gas molecular wt (g/m) (29.0 at STP)

d) Calculate Brink flow rate:

$$Q_B = 0.0519\ (\Delta P_E)^{0.44} \tag{4-3}$$

where

Q_B = Brink flowrate (cfm)

e) Calculate stack velocity based on Brink flow rate:

$$V_{SB} = \frac{Q_B}{(5.07 \times 10^{-4})\ D_B^2} \tag{4-4}$$

where

V_{SB} = Brink stack velocity (ft/sec)

D_B = Brink nozzle diameter (mm)

f) Determine percent isokinetic (I):

$$I = \frac{V_{SB} - V_S}{V_S}\ (100\%) \tag{4-5}$$

g) Make a continuous plot of I on x-y graph with I on the y-axis and the day on the x-axis. This daily plot should be kept as a permanent record of the Brink runs. As the data begins to accumulate trends will be established. Normally I should vary randomly between 90 and 110%. Consistently high or low I values indicate systematic errors in sampling and call for a review of procedures and equipment.

Errors can be due to:

- Equipment - Inaccurate or malfunctioning magnehelic gauges, dry test meter or thermocouples. Refer to Tables 7 and 8 for troubleshooting and calibration procedures.

- Data recording - Wrong numbers taken or misplaced on the Field Data Sheet. Double check all entries.

- Calculations - Either the wrong data were input input or a mathematical error was made. Have different individuals do the calculation.

2. Stack Mass Loading Evaluation

Since an Aerotherm (Method 5) mass reading (C_A) is performed prior to the Brink run, it is possible to obtain an approximate comparison of Brink mass loading values (C_B) with those obtained from the Aerotherm.

a) Correct the Aerotherm dry meter volume (V_m) to standard conditions:

$$V_m(STD) = V_m \left(\frac{528}{\bar{T}_m+460}\right)\left(\frac{\bar{P}_m}{P_{STD}}\right) \qquad (4\text{-}6)$$

where

$\bar{T}_m$ = Average meter temperature (^oF)

$\bar{P}_m$ = Average absolute meter pressure (in. Hg)

P_{STD} = Standard pressure (29.92 in. Hg)

b) Determine Aerotherm mass loading (C_A) in grains/scf:

$$C_A = \frac{W_{TA}\ (15.43)}{V_m(STD)} \qquad (4\text{-}7)$$

where

W_{TA} = Total particle weight recovered from Aerotherm train (grams)

c) Using Brink particle size computer program, calculate the actual volume of air that passed through the impactor.

d) Correct this value to standard conditions:

$$V_{B(STD)} = V_{B(ACT)}\ (1 - B_W)\ \left(\frac{528}{T_I+460}\right)\left(\frac{P_{IA}}{P_{STD}}\right) \qquad (4\text{-}8)$$

where

B_W = Volume fraction of H_2O in gas sample (obtained from mass loading run)

$V_B(ACT)$ = Volume sampled by Brink (acf)

$V_B(STD)$ = Volume sampled by Brink (dscf)

e) Calculate mass loading for Brink (C_B) in grains/scf:

$$C_B = \frac{W_{TB}\ (15.43)}{V_{BC(STD)}} \qquad (4\text{-}9)$$

where

W_{TB} = Weight collected from Brink train (probe, cyclone, stages, and filter in grams).

f) Ratio calculated mass loadings

$$G = C_B/C_A \qquad (4\text{-}10)$$

where

G = correlation variable for Brink and Aerotherm system

g) Plot the daily values and observe the trend. The expected range for G should be 0.7 to 1.3. Consistently high G values may indicate contamination or incorrect flow rate calculations. Verify that the correct values are used in the Brink program. Also check the calibration of the magnehelic gauges. Consistently low G values are more likely to be found. The most probable cause for this result in poor overall particulate matter recovery, but especially poor recovery from the probe. Review cleaning procedure and make extra effort to clean the probe and connecting tubing properly.

3. Fine Particle Mass Loading Evaluation

The previous two sections have described evaluation methods that measure the quality of sampling and the overall sample recovery of the test crew. The efficiency of the impactor can be monitored by determining the fine particle mass loading. The fine particle mass loading for the Aerotherm system is defined as the weight of the particulate matter found on the filter divided by $V_{M(STD)}$. For the Brink system the fine particle grain loading is the weight of particulate matter found after the first stage divided by the $V_{B(STD)}$. Comparison of these values will provide an indication of the operation of the impactor, since all material after the first stage should be localized on the stages and not require rinsing operations. The evaluation procedure follows:

a) Determine the Aerotherm fine particle mass loading (C_{FA}) in grains/scf:

$$C_{FA} = \frac{W_{FA}\ 15.43}{V_{M(STD)}} \tag{4-11}$$

where

W_{FA} = Particulate matter weight on Aerotherm filter (g)

b) Determine Brink fine particle mass loading (C_{FB}) in grains/scf:

$$C_{FB} = \frac{W_{FB}\ 15.43}{V_{B(STD)}} \tag{4-12}$$

where

W_{FB} = Particle weight found in Brink system after the first stage (g)

c) Determine fine particle ratio (G_F):

$$G_F = \frac{C_{FB}}{C_{FA}} \tag{4-13}$$

d) Plog G_F daily and observe any trends. The expected range for G will be from 0.7 to 1.3. High results (G>1) can be from:

- Low flow rate values for the Brink systems - check calculations and calibration of magnehelic gauges.

- Contamination - Review laboratory procedures (Section 4.1)

Low results on (G<1) are the more probable and can be due to:

- Loss of material on transfer - review handling procedures.
- High flowrate values - check calculations and magnehelic gauge calibration.
- Grease weight loss - verify impactor temperature was less than 347°F. Note any amber discolorate on filter signifying grease flow through. Correct these problems by controlling temperature to <347°F, placing a thin film of grease on the plates and maintain gas flow to ≤0.08 acfm.

4.2.2 MRI Methodology

The impactor selected to measure the particle size distribution exiting the wet scrubber system is the MRI impactor. The MRI impactor system consists of a 1/2-inch Aerotherm probe connected directly to the impactor (Figure 13). The MRI impactor is designed to measure the aerodynamic size distribution between 30 and 0.3 microns distributed over six stages and a final filter. With Viton O-rings and Apiezon H greased stages, the maximum operation temperature is 175°C (347°F) at a maximum flowrate of 0.8 acfm.

In this application the MRI system is used as an out-of-stack extractive sizing method. Using an Aerotherm probe, a velocity profile for the duct is obtained. The average velocity is calculated and used to select a nozzle that will sample at the average isokinetic velocity but at or below a flow rate of 0.8 acfm.

Temperature control of the impactor system is maintained by monitoring the stack and outlet gas temperature from the impactor. The necessary heat is supplied by a specially designed Glass-Col heating mantle. The gas flow rate is monitored by measuring the ΔH across calibrated orifice with a magnehelic gauge.

The amount of material collected is determined by weighing the collection stages before and after the run. Probe rinses are added to the first stage. The collection plate and filter are thermally conditioned and desiccated prior to weighing. After sampling, the samples are desiccated to constant moisture content prior to re-weighing. Because of the potential for systematic errors in weighing, blanks consisting of spare collection stages and filters are conditioned and weighed along with the samples to monitor weighing errors (see Section 4.1.8).

a) Critical Checkpoints

Table 5 is a checklist of critical items that should be followed during the test run. These critical items consist of:

- Recommended flow rates, temperatures, and sampling times
- Reminders on laboratory and sampling technique
- Specific equipment checks.

While this list is provided for review prior to the sampling run, its best use is an an on-site checklist for the supervisor and quality assurance personnel during the run. During a test audit the supervisor or QA representative should initial each item successfully completed. The entire list should be included with the documentation of that test run. The operating personnel might also like to have copies of the checklist for reference during the execution of the test run. Copies should be posted in the laboratory and sampling site for this purpose.

b) Data Monitoring Procedures

Dry aerosol sampling procedures cannot be tested in the classical fashion, i.e., with spiked samples, to determine their reliability, but there are simple monitoring activities that can be carried out on a daily basis. These activities include:

- Calculation of percent isokinetic sampling which provides information on the quality of sampling.
- Comparison of Aerotherm/MRI grain loadings which determines the efficiency of particle recovery from system.

The following paragraphs detail these procedures.

1. Isokinetic Sampling Tests

Isokinetic sampling is sampling at a rate (measured at the probe nozzle) equal to the velocity of the gas flowing by the probe. Unless gas flows are sampled isokinetically, larger or smaller particles can be preferentially collected depending on whether the sample rate is less than or

TABLE 5. CRITICAL CHECKPOINTS FOR MRI DRY AEROSOL SYSTEM

Checkpoint	Initials		Remarks
	Supervisor	QA Inspector	
I. Conditioning and Preweighing Procedures:			
A. Conditioning			
- Plates cleaned with toluene and NaOH prior to greasing.			
- Hands gloved.			
- Thin film of grease applied to plate.			
- Handle clean filter with tweezers.			
- Clean petri dishes and watch plates prior to storing impactor plates and filter.			
- Check labeling system on petri dishes.			
- Plates conditioned at 175°C for 2 hours.			
- Filter conditioned at 290°C (±10°C) for 4 hours.			
- Desiccant fresh (color B/P?)			
- Plates and filter desiccated for 2 hours.			
B. Weighing			
- Balance area clean.			
- Balance pan clean.			
- Balance leveled.			
- Balance zeroed.			
- Weigh standard.			
- Weigh impactor plate and filter blanks for each set of samples.			
- How long were petri dishes and impactor stages left open to lab air (min.)?			
- Data recorded on correct data sheets.			
- Note condition of plates (color of grease, thickness of coating, etc.) on data sheet.			
II. Laboratory Impactor Preparation			
- Plates and filter identifications and weights recorded.			
- Impactor inspected for wear (threads, jet plates, inlet nozzle, O-rings)			
- Impactor cleaned.			
- Work area cleaned and bench covered with Whatman paper.			
- Hands gloved.			
- Impactor stages loaded, starting from last stage.			

-Continued-

TABLE 5. CRITICAL CHECKPOINTS FOR MRI DRY AEROSOL SYSTEM (CONTINUED)

Checkpoint	Initials		Remarks
	Supervisor	QA Inspector	
- Backing, filter, stainless steel seal washer placed in the filter stage.			
- Perform impactor leak test.			
- Inlet tubing sealed off to prevent particles from entering impactor.			
III. Site Set-Up			
- Impactor maintained in vertical position during transfer to site.			
- Brush inside probe prior to run.			
- Rinse probe with acetone until rinse solution is clear.			
- Fresh solutions placed in impingers.			
- Leak test performed and magnehelic gauges zeroed.			
- Leak rate must be less than 0.02 cfm.			
- Thermocouple leads attached to impactor			
- Skin temperature controlled to <347°F.			
IV. Sampling Run			
- MRI gas out temperature maintained at highest stack reading +25°F.			
- MRI gas out temperature must never exceed 347°F.			
- Select flowrate below 0.8 cfm.			
- Select sampling time to collect no more than 10 mg on any stage.			
- Check seal between probe and port to prevent any outside air entering the stack.			
- Impactor carried in an upright position to laboratory.			
- Support equipment cleaned prior to next run.			
- Report any experimental problems or unusual occurrances on data sheet.			
V. Sample Recovery			
- Keep probe in a horizontal position prior to particulate matter recovery.			
- Keep impactor upright while transferring to lab.			
- Particulate matter from probe rinsed into 250 mL Erlenmeyer flask.			
- Probe and tubing connections inlet to the impactor are brushed and rinsed with acetone until rinse stream is clean. Rinsings collected in Erlenmeyer flask and saved for weighing in a tared 50 mL beaker.			

-Continued-

TABLE 5. CRITICAL CHECKPOINTS FOR MRI DRY AEROSOL SYSTEM (CONTINUED)

Checkpoint	Initials		Remarks
	Supervisor	QA Inspector	
- Transfer probe and tubing washes from the Erlenmeyer to tared 50 mL beaker.			
- Estimate and record any loss of material during transfer to tared 50 mL Erlenmeyer (% lost___).			
- Dry probe washings in oven at 110°C for 1 hour.			
- Use gloves during impactor stage removal procedure.			
- Impactor cooled to room temperature			
- Use tweezers to remove impactor plates.			
- Inspect impactor walls and jet nozzles for particulate matter. Brush any particulate matter there onto the impactor plate. Note presence of particulate matter on the walls or jet nozzles on data sheet.			
- Inspect filter holder for pieces of filter material.			
- Collect all pieces of filter material from filter holder and place them with the intact filter for weighing.			
- Keep all exposed impactor plates and samples covered.			
- Desiccate all samples 2 hours prior to weighing.			
- Correct all weights for any change in impactor and filter blank weights.			
- Note tackiness of plates.			
VI. Data Analysis Verification			
- Plot Aerotherm and MRI grain loading values on a daily basis (Y-axis for grain loading, X-axis day).			
- Plot the daily percentage isokinetic for MRI runs (Y-axis for percent isokinetic, X-axis day)			
- Plot sample blank weight change (Y-axis wt., X-axis day).			

greater than the stream flow rate, respectively. The closer to isokinetic conditions, the more representative the particle sample will be. Thus, the degree of isokinetic sampling is an expression of the quality of the sampling runs. The normal criteria of acceptability is ±10 percent of the correct isokinetic flow rate.

Because the internal gas velocity determines the size cutoffs for the collection stages, the impactor must be run at one flow rate if only one size range of particles is to be deposited on a given stage. Consequently the velocity profile is determined just prior to the test run and the impactor is run at the average isokinetic flow rate.

The procedure for the isokinetic check follows:

(a) From the actual velocity profile measured during the run compute the average $\overline{\sqrt{\Delta P}}$.

(b) Calculate the average stack velocity:

$$\overline{V}_S = (85.48)\ C_P\ \overline{\sqrt{\Delta P}} \left(\frac{\overline{T}_S + 460}{P_S\ M_S}\right)^{1/2} \qquad (4\text{-}14)$$

where

$\overline{V}_S$ = Average stack velocity (ft/sec)

C_P = Pitot tube coefficient

$\overline{\sqrt{\Delta P}}$ = Average square root of the velocity head (in. $H_2O)^{1/2}$

$\overline{T}_S$ = Average stack temperature (°F)

P_S = Absolute stack gas pressure (in. Hg)

M_S = Stack gas molecular weight

(c) Correct MRI meter volume (V_{Mm}) to flow rate at stack conditions:

$$Q_{SM} = V_{Mm}\ (1 + B_w) \left(\frac{\overline{T}_S + 460}{\overline{T}_m + 460}\right) \left(\frac{\overline{P}_m}{P_S}\right) \left(\frac{1}{t}\right) \qquad (4\text{-}15)$$

where

Q_{SM} = MRI flow rate (acfm)

V_{Mm} = MRI dry test meter volume (cf)

B_w = Volume fraction of water in gas stream obtained from previous mass loading run

$\overline{T}_S$ = Average stack temperature

$\overline{T}_m$ = Average meter temperature

$\overline{P}_M$ = Average absolute meter pressure (in. Hg)

P_S = Absolute stack pressure (in. Hg)

t = Sampling time (min)

(d) Calculate stack velocity based on MRI flow rate:

$$V_{SM} = \frac{Q_M}{0.327\ (D_M)^2} \qquad (4\text{-}16)$$

where

V_{SM} = MRI stack velocity (ft/sec)

D_M = MRI nozzle diameter (in.)

(e) Determine percentage isokinetic (I):

$$I = \frac{V_{SM} - V_S}{V_S}\ (100\%) \qquad (4\text{-}17)$$

(f) Make a continuous plot of I on x-y graph with I on the y-axis and the day on the x-axis. This daily plot should be kept as a permanent record with the MRI runs. As the data begins to accumulate, trends will be established. Normally the points should vary. Consistently high or low I values indicate systematic errors in sampling and call for a review of procedures and equipment. Errors can be due to:

- Equipment - Inaccurate or malfunctioning magnehelic gauges, dry test meter or thermocouples. Refer to Tables 7 and 8 for troubleshooting and calibration procedures.

- Data recording - Wrong numbers taken or misplaced on the Field Data Sheet. Double check all entries.
- Calculations - Either the wrong data were input or a mathematical error was made. Have different individuals do the calculation.

2. Stack Mass Loading Evaluation

Since an Aerotherm (Method 5) mass reading is performed prior to the MRI run, it is possible to compare MRI mass loading values (C_M) with those obtained from the Aerotherm (C_A).

(a) Correct the Aerotherm dry test meter volume (V_m) to standard condition:

$$V_{m(STD)} = V_m \left(\frac{528}{\overline{T}_m + 460}\right)\left(\frac{\overline{P}_m}{P_{STD}}\right) \qquad (4\text{-}18)$$

where

$\overline{T}_m$ = Average meter temperature ($^{\circ}F$)

$\overline{P}_m$ = Average absolute meter pressure (in. Hg)

P_{STD} = Standard pressure (29.92 in. Hg)

(b) Determine Aerotherm mass loading (C_A) in gr/scf:

$$C_A = \frac{W_{TA}\,(15.43)}{V_{M(STD)}} \qquad (4\text{-}19)$$

where

W_{TA} = Total particle weight recovered from Aerotherm train (g)

(c) Using V_{Mm} and equation 4-18, calculate the MRI gas volume sampled at standard conditions $\left[V_{M(STD)}\right]$

where

V_{Mm} = Dry test meter reading during MRI run

(d) Calculate mass loading for MRI (C_M) in gr/scfm:

$$C_M = \frac{W_M \, (15.43)}{V_{M(STD)}} \qquad (4\text{-}20)$$

where

W_M = Weight collected from MRI train (probe, stages, and filter in g)

(e) Ratio calculated mass loadings:

$$G = C_M/C_A \qquad (4\text{-}21)$$

where

G = Correlation variable for MRI and Aerotherm system

(f) Plot the daily G values and observe the trend. The range of acceptable agreement is from 0.7 to 1.3. Consistently high G values indicate contamination or incorrect flow rate calculations. Verify that the correct values and equations were used. Also check the calibration of the magnehelic gauges. Consistently low G values are more likely to be found. The most probable cause for this result is poor overall particulate matter recovery, but especially poor recovery from the probe. Review cleaning procedures and make extra effort to clean the probe and connecting tubing properly.

Other reasons for low G values are:

- High flow rate values - check calculation and dry test meter calibration.
- Grease weight loss - verify impactor temperature was less than 347°F. Note any amber discoloration on filter signifying grease flow through. Correct these problems by controlling temperature to <347°F, placing a thin film of grease on the plates, and maintaining gas flow at ≤0.8 acfm.

4.2.3 Goksoyr-Ross Methodology

At a given temperature a gas can hold a specific amount of a liquid as a vapor. As the temperature is lowered less of the liquid can exist as a gas and condensation begins. The amount of liquid existing as a gas at a given temperature will be related to the liquid's boiling point. Consequently various liquids will condense at different temperatures. Thus a flue gas can be conditioned to a specific temperature to separate H_2SO_4 (b.p. $\sim 300^oC$) from water (b.p. 100^oC).

The G/R system (Figure 19) consists of a quartz probe heated to 316°C (600^oF) to collect the gas from the duct. No attempt is made to sample isokinetically. The flow rate is controlled during the sampling by monitoring the total flow at the dry test meter with a stopwatch. The gas stream then passes into a heated (288^oC-550^oF) quartz filter holder which contains a Tissuequartz filter to remove particulate matter from the gas stream. The filter temperature must be maintained to ensure quantitative recovery of H_2SO_4. The clean flue gas then flows into the water jacketed coil maintained at 60^oC (140^oF) to condense and collect any sulfuric acid vapor that might be present in the gas stream. Temperatures dropping below this value will condense H_2O and SO_2 and cause positive errors. After a period of time (1 hour or until 1/2 to 2/3 of the coils are frosted), the coil is rinsed out and the acid determined by titration with NaOH using bromophenol blue as the indicator. For a discussion of the proper procedures to be used during this titration refer to 4.1.9.

a) Critical Checkpoints

Table 6 is a checklist of critical items that must be followed during the test run. This critical item list consists of:

- Recommended flow rates, temperatures and sampling times
- Reminders on laboratory and sampling techniques
- Specific equipment checks.

While this list is provided for review prior to the sampling run, its best use is an an on-site checklist for the supervisor and quality assurance personnel during the run. During a test audit the supervisor or QA representative should initial each item successfully completed. The entire

TABLE 6. CRITICAL CHECKPOINTS FOR G/R H_2SO_{40} SAMPLING SYSTEM

Checkpoint	Initials		Remarks
	Supervisor	QA Inspector	
I. Laboratory Preparation			
- Inspect and clean G/R coil. Both filter holder and G/R are cleaned with hot chromic acid solution and D.I. H_2O.			
- Rinse with acetone and air dry G/R coil.			
- Place Tissuequartz filter in filter housing.			
- Check seal between end of joint and filter.			
- Do not use grease on joints.			
- Inspect and clean all glass joints.			
II. Site Set-Up			
- Rinse the inside of probe prior to run.			
- Rinse probe with acetone until rinse solution is clear.			
- Perform leak test.			
- Leak rate must be less than 0.003 cfm or 80 mL/min.			
- Zero Magnehelic gauges.			
- Thermocouple leads attached to probe and filter.			
- G/R water bath held at 140°F (±2°F)			
- Leak test train.			
- Probe temperature maintained at 600°F (±30°F).			
- Gas temperature out of filter holder held at 550°F (±10°F)			
- Fresh solutions placed in impingers.			
- Fresh absorbant replaced in final impinger.			
- Adjust flowrate in system to 10 Lpm.			
III. Sampling Run			
- Turn vacuum pump on just before inserting probe in the stack.			
- Check seal between probe and port to prevent any outside air entering the stack.			
- Run test for 1 hour or until coils are frosted to 1/2 to 2/3 of their length.			
- After run cap both ends of the probe and lay in horizontal position.			
- Rinse the G/R coil into the modified Erlenmeyer flask with a maximum of 40 mL of D.I. H_2O.			
- Was any of the solution lost (mL estimated)?			
- Handle hot glassware carefully to prevent personnel injury and damage to equipment.			

-Continued-

TABLE 6. CRITICAL CHECKPOINTS FOR G/R H_2SO_{40} SAMPLING SYSTEM (CONTINUED)

Checkpoint	Initials		Remarks
	Supervisor	QA Inspector	
- After probe has cooled, the probe is rinsed with a maximum of 40 mL. D.I. H_2O into a 125 ml Erlenmeyer.			
- Was any solution lost (mL estimated)?			
- Clean support equipment prior to next run.			
- Save filter for titration.			
IV. Laboratory Analysis			
- Clean glassware prior to titration.			
- Use Bromphenol Blue indicator.			
- Is the NaOH buret protected with a CO_2 absorbant tube?			
- When was NaOH standardized last (Date)?			
- Filter any solution that has suspended particulate.			
- Use same number of indicator drops for each titration.			
- Perform indicator blank on a volume of D.I. H_2O equal to sample aliquot used.			
- Indicator blank added to H_2SO_4 milli-equivalents found.			
- Perform all analyses in triplicate.			
V. Data Analysis Verification			
- Obtain and titrate test samples from main laboratory.			
- Plot daily inlet and outlet H_2SO_4 values (Y-axis for ppm H_2SO_4, X-axis for day)..			

list should be included with the documentation of that test run. The operating personnel might also like to have copies of the checklist for reference during the execution of the test run. Copies can be posted in the laboratory and sampling site for this purpose.

b) Data Monitoring Procedures

The data monitoring procedures for the G/R system are devoted mainly to the acid-base titration performed in the laboratory and to the monitoring of the H_2SO_4 ppm values calculated.

1. Acid Base Titration

In order to check the accuracy of the titrations performed on the G/R samples, an independent check of the NaOH solution and titration method is required. From main laboratory or an independent laboratory, a standardized sample of H_2SO_4 approximately 0.01 N should be analyzed by the trailer personnel every couple of weeks. Analysis of the sample should be triplicate and reported to 3 places (0.X Y Z). Analysis of this sample will provide information on the precision of the G/R titrations and accuracy of the results.

The procedure follows:

(a) Take a 10 ml aliquot of the unknown standard

(b) Titrate in triplicate with Bromophenol Blue to the blue end point and record the number of milliliters used.

(c) Determine the normality of the solution from:

$$N_A = \frac{N_B V_B}{V_A} \qquad (4\text{-}22)$$

where

N_A = Normality of the acid

V_A = Volume acid aliquot taken (mL)

N_B = Normality of the base

V_B = Volume of the base used to titrate the sample (mL)

The results of the determination should not differ by more than ±10% within the triplicate numbers nor should the determined normality be off by more than ±10%.

If the values differ by more than 10%:

- Check the calculations and be sure the correct values have been used
- Repeat the analysis
- If the value is still off, restandardize the NaOH with KHP.
- Repeat the test.

2. Data Monitoring by Statistical Quality Control

Since there is a direct correlation between the inlet and outlet values such that the outlet is predictable from the inlet contamination, a simple control chart using regression analysis can be used Figure 25. Simply plot all the values obtained on this chart. The region between the -2σ and $+2\sigma$ limits should contain, in the long run, 95% of all future paired measurements.

The σ limits are based on 44 of paired SO_3 measurements completed at the Venturi scrubber. It is assumed that similar results will be obtained on the TCA so that this chart can be used for both systems. The warning limits will be the (90,90) limits. That is, it is expected that 90% of the future observations will lie within such limits, 90% of the time. The rejection limits, or the limits that indicates that the system is out of control will be the (90,95) limits. That is, it is expected that 95% of the future observations will be within these limits 90% of the time. As trends develop, data that is widely outside of the normal range can be spotted. When those events occur, be sure to:

- Record any unusual occurrences during the test on the data sheets

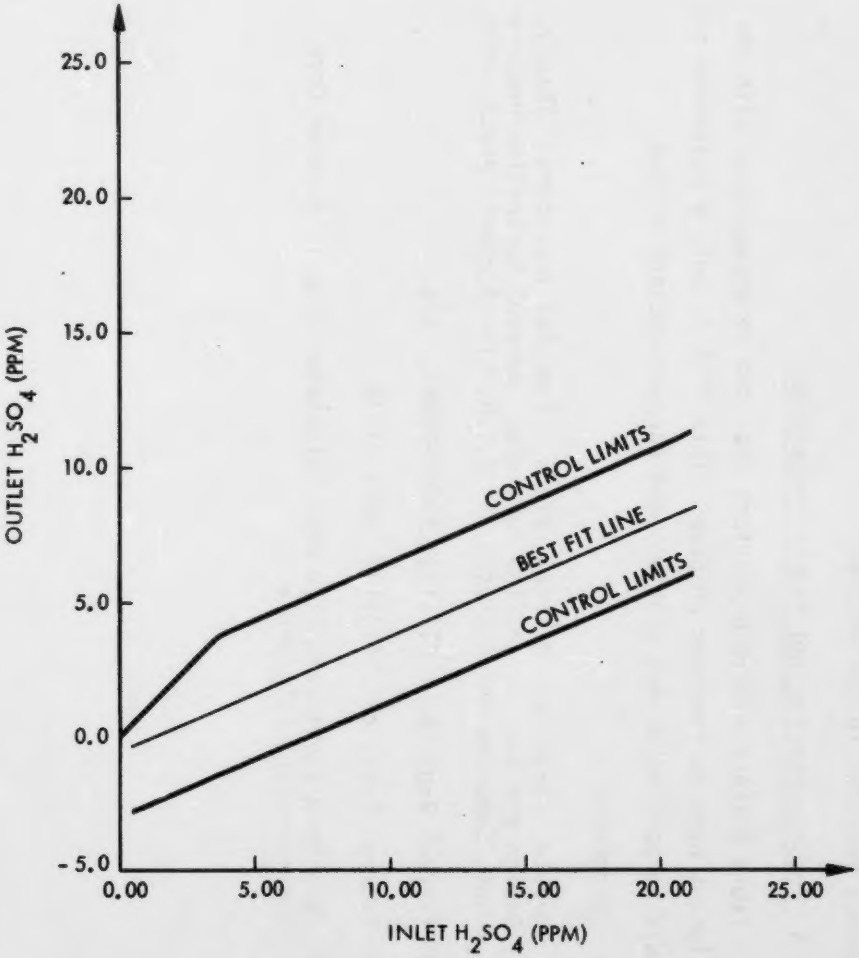


Figure 25. Control Chart for Controlled Condensation Measurements of H_2SO_4

- Check with the power plant of scrubber control room to find out if any mechanical problems occurred during the run.
- Verify that all the laboratory numbers are correct and repeat the analysis if any solution is left over.

4.2.4 Maintenance Schedules

Table 7 details the recommended maintenance schedule. Following of this schedule is imperative to prevent breakdown and to maintain the high accuracy required in the program.

4.2.5 Troubleshooting and Repair Procedures

Table 8 lists possible problems that can be encountered with the equipment used in the test program. This list is only a beginning and should be updated as new problems are encountered and solved.

4.3 REFERENCES

4-1 Marple, Virgil and Willeke, Klaus. Inertial Impactors: Theory, Design and Use. From Fine Particle: Aerosol Generation Measurement, Sampling and Analysis, ed B.Y.H. Liu, Academic Press, 1976.

4-2 Federal Register. 41(111): 23082-23083, 1976

4-3 Federal Register. 41(11): 23076, 1976

4-4 Aerotherm Isokinetic Flow Rate Calculator Manual, Accurex Corp. Mountainview, California

TABLE 7. GENERAL MAINTENANCE SCHEDULE

Component	Maintenance Schedule By:			Calibration Procedure
	Run	Week	Month	
S-Pitot nozzles	Inspect alignment after each run from head-on and side angles.	Brush-out inside of pitot tubes	Calibrate C_p every three months	Send to Muscle Shoals for C_p check and calibration.
	Blow out particulate matter after each run.			
Probe nozzles	Inspect nozzle for damage			Measure nozzle ID with micrometer.
	Brush nozzle before and after run to remove inside particulate.			
	Measure ID with micrometer before each run.			
	Check alignment of nozzle before each run by looking at nozzle from head-on and side angles.			
Steel probes	Before and after each run, brush and rinse with reagent grade acetone or Freon until rinse is clean.	Use wire bush to clean inside surface of the probe.		N/A
Quartz probe	Before and after each run brush and rinse with reagent grade acetone or Freon until rinse is clean.			N/A
Impingers	Rinse out after each run with DI water.			N/A
	Inspect and clean seal area and O-rings.			
	Leak test before each run.			Leak test at 380 torr (15" Hg) and verify that a leak rate of <0.3 lpm (0.02 cfm) is maintained.

-Continued -

TABLE 7. GENERAL MAINTENANCE SCHEDULE (CONTINUED)

Component	Maintenance Schedule By:			Calibration Procedure
	Run	Week	Month	
Pump	Before each run check leak rate in pumping system.		Inspect vanes on diaphragm. Inspect and clean motor brushes.	Depending on the system, a leak rate must be less than a certain value. See specific critical checkpoint table for information on recommended maximum leak rates.
Swagelok fittings	Inspect fitting, especially ferrule and seat for wear and dirt. Clean or replace fitting as required.			N/A
Brink Impactor	Clean impactor Check washers for wear. Inspect jets for blockage.		Every 3 months check impactor flowrate calibration.	Hook to dry test meter, start flow, time, read Δp across impactor. Record data. Repeat at different flowrate. Compare results to calibration chart.
			Every 6 months check D_{50} calibration.	Send impactor to Southern Research Institute in Birmingham, Alabama for calibration.
MRI Impactor	Clean impactor after each run. Note: Acetone should not contact Viton O-rings. Check O-rings for wear Inspect jet nozzles for blockage.		Every 6 months check D_{50} calibration.	Send impactor to Meteorology Research, Inc., Altadena, Cal. for calibration.
CCS filter holder	Inspect and clean after each run. Replace filter after each run.	Clean frit each week in hot chromic acid for 12 hours. Rinse to neutral pH with DI H_2O.		N/A
CC coil	Inspect and clean after each run	Clean coils and frit each week in hot chromic acid for 12 hours. Rinse to neutral pH with DI H_2O.		N/A
NaOH solution		Standardize the NaOH with KHP weekly.		

-Continued-

TABLE 7. GENERAL MAINTENANCE SCHEDULE (CONTINUED)

Component	Maintenance Schedule By:			Calibration Procedure
	Run	Week	Month	
Thermocouples	Inspect lines for wear and kinks.	Clean tips of shielded TCs Clean connector prongs with steel wool.	Calibrate thermocouples	Calibrate TC at two points (ice-water and near boiling). Compare TC readings to mercury thermometer. Replace TC if agreement is not within 3°C (6°F).
Temperature indicator	Clean readout of all dust.		Have electronics shop remove the back and clean the inside of the unit.	Perform thermocouple calibration with readout unit using independently calibrated thermocouple.
Oven or probe heaters			Check indicated temperature with calibrated thermocouple.	Check indicated temperature readings with calibrated thermocouple.
Connecting lines	Blow out connecting lines with air Visually inspect exterior for wear. Especially inspect hose to fitting connections.	Flush with water and dry with clean plant air.		N/A
Magnehelic gauges	Check lines to gauges to ensure there is no blockage. Zero gauge before run with both ports open to the atmosphere.		Every month check calibration of gauge versus water or mercury manometer.	Calibrate versus water or mercury manometer depending on the range of the gauge. Connect manometer and gauge to vacuum or pressure source simultaneously using a tee. Check the magnehelic gauge's readings at low, medium and high points in its range.
Calibrated orifice		Clean and inspect critical orifice	Calibration check	Calibration procedure for a critical orifice is found on page A6 in Appendix A.
Dry test meter	Clean exterior		Calibrate versus wet test meter every 3 months	Run wet and dry test meters in series, note temperature and pressure. If dry test meter is >3% off, send to factory for recalibration.

TABLE 8. TROUBLESHOOTING AND REPAIR

Component	General Remarks	Problem	Repair Sequence
S-Pitot Nozzles	Alignment of pitot tubes is critical. The tubes must be facing 180° with respect to each other and parallel to gas flow in the duct.	Misaligned nozzle	Return S-Pitot tubes to original 180° alignment. Align nozzles to be parallel to gas flow. Position face of nozzle to be perpendicular to gas flow.
		Damaged edge	File and buff edge to smooth oval - repeat alignment checks.
Probe Nozzles	A smooth circular edge is required for accurate sampling. Alignment of nozzle face must be perpendicular to gas flow.	Nozzle wear or damage	File and buff edge to smooth circle.
		Misalignment	Loosen Swagelok fitting and realign (x-axis) nozzle face to perpendicular to gas flow. Bend nozzle neck (y-axis) so that nozzle face is perpendicular to gas flow.
Steel Probe	Because the probe contains the S-pitot nozzles, alignment of the probe must be checked with a level once the probe is in the stack.	Normal wear and cleanliness	Pitting on the inside of the probe should be removed by use of a wire brush.
Quartz Probes	Avoid mechanical shocks especially when probe is hot. Before cleaning probe with liquids, allow the probe to cool to air temperature.	Normal wear and cleanliness	Brush and rinse with acetone after each run (Note: Test brush to insure it is not dissolved by the acetone).
Impingers	Impingers should be cleaned with soap and water. Deposits should not be allowed to build up inside impinger. All nozzles should reach to within ±1.3 cm (0.5") of the bottom of the impinger. To insure good seals, keep the impinger seals clean.	Normal wear and cleanliness	Rinse out with DI water after each use. Dry impinger to be used for moisture trap. Clean sealing edges and O-ring of impinger.

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TABLE 8. TROUBLESHOOTING AND REPAIR (CONTINUED)

Component	General Remarks	Problem	Repair Sequence
Impingers		Leakage in impinger system	Check all Swagelok fittings. Inspect impinger seal area for dirt or damage. Clean area if dirt found. Use larger O-ring. If all other measures fail to locate leak, pressurize and immerse in water to find leak.
Thermocouples	Thermocouple (TC) leads and wire are fragile and require care in arranging the equipment set-up to prevent kinking and stripping of leads. Never pull a TC apart by pulling on the lead. Verify that the polarity is not reversed anywhere in the system. Be sure that the same type of TC wire and connectors are used in the system (Iron-constantan or chromel-alumel). Do not bend casing of shielded thermocouple.	Temperature indicator fluctuating over wide range.	Locate possible short in TC wire or connectors. Once portion of wire with short is located, mark and have the wire replaced. Have readout checked by electrical shop if no external short can be found.
Temperature Indicator	Store in dust free area	Temperature readings fluctuating on one channel.	Check thermocouple for short in lead or connectors.
		No temperature readout or fluctuating temperatures on all the channels with thermocouples attached.	Return to electronic shop for repair. Return to manufacture if problem cannot be found.
Oven or probe heaters	Never exceed maximum temperature as stated in the manufacturer's manual.	No temperature rise with current on.	Check electrical connections. Check main power. Check fuses and circuit breakers. Verify thermocouple connected.

-Continued-

TABLE 8. TROUBLESHOOTING AND REPAIR (CONTINUED)

Component	General Remarks	Problem	Repair Sequence
Connecting lines	While these lines are either heavy vacuum hose or steel braided Teflon lines, care should be taken to minimize weight supported by the lines and excessive mechanical abuse. The Aerotherm lines should never be kinked to cut off flow.	General maintenance.	Replace any worn or corroded parts.
Magnehelic gauges	Magnehelic gauges measure the pressure differences felt by an internal diaphragm. The diaphragm is magnetically linked to the display needle. These gauges can stand a certain amount of over-pressure, but should not be left in that condition for long. The normal operating temperature is 30 to 140°F.	Pegged needle	Remove leads, blow into both sides and reset zero if necessary.
		No reading	Check connections to gauge. Check leads for blockage. Clean lines if necessary.
		Erratic Readings	Fluctuation in pressure reading probably due to surges or cycles in pumping system. Place Swagelok snubber on the inlet to the gauge.
Calibrated Orifice	A calibrated orifice is a constriction in a tube in which a gas is flowing that causes a difference in pressure between the upstream and downstream sides of the constriction. This pressure differential (ΔH) is related to the rate of flow.	Higher ΔH values for flowrates at the same conditions.	Recheck ΔH calculation. Check lines for particulate matter. Inspect critical orifice for corrosion or blockage. Clean orifice with copper wire. Recalibrate orifice.

-Continued-

TABLE 8. TROUBLESHOOTING AND REPAIR (CONTINUED)

Component	General Remarks	Problem	Repair Sequence
Pump	Care must be taken in shutting the pump off after a run. Rapid shutdown without bleeding air into the pumping system will cause the impingers to back-up towards the filter.	Leakage (oil-less)	Check all valve and hosing connections leading to pump. If the leakage has been isolated in the pump, disassemble pump and inspect vanes for wear and replace if necessary.
		Leakage (diaphragm)	For leakage or low flow in diaphragm pumps check the diaphragm cover to ensure it has not vibrated loose. Remove face plate and inspect diaphragm for signs of wear or pinholes. Check the diaphragm gasket for wear, replace if necessary.
Swagelok fittings	Swagelok fittings are designed to seal with a minimum of tightening. Excessive torque applied to the fitting will eventually cause leakage.	Installation	Insert the tubing in the service. Insert the tubing into the Swagelok tube fitting. Make sure that the tubing rests firmly on the shoulder of the fitting and that the nut is finger-tight. Due to the variation of tubing diameters, a common starting point is desirable. Therefore, use a wrench to snug up the nut until the tubing will not turn (by hand) in the fitting. At this point, scribe the nut and body at the 6 o'clock position of the fitting. Now while holding the fitting body steady with a backup wrench, tighten the nut one-and-one-quarter turns. Watching the scribe mark, make one complete revolution and continue to the 9:00 o'clock position.

-Continued-

TABLE 8. TROUBLESHOOTING AND REPAIR

Component	General Remarks	Problem	Repair Sequence
Swagelok Fitting		Reinstallation	Tubing with preswaged ferrules inserted into the fitting until front ferrule seats in fitting. Tighten nut by hand. Rotate nut about one-quarter turn with wrench (or to original one-and-one-quarter tight position) then snug slightly with wrench.
Dry Test Meter	These meters are very sensitive to mechanical shock and should be handled with care. Corrosive gas from the stack should never be passed through the meter without prescrubbing.	Incorrect volume readings.	Check meter for blockage Check mechanical linkage for wear Recalibrate meter
Brink impactor	The Brink impactor operates at a very low flowrate which requires that very low leak rates must be maintained. Any interior part must never be cleaned with any material that can scratch the metal.	Leakage > 80 ml/min	Check all fitting and connections Tighten impactor housing Check all Teflon seals. Replace if necessary
		Plugged nozzle	Use copper wire to dislodge material After cleaning, check nozzle size.
MRI impactor	The MRI impactor has an aluminum housing which requires care to prevent the thread from being stripped. Since the jet plates are removed during sample recovery, care must be taken to ensure that the plates are not scratched.	Leakage >0.02 cfm	Check all fittings Inspect O-ring seals for damage or flattening. Replace worn O-rings Tighten impactor housing Replace standard O-rings with Viton oversized O-rings
		Melted O-ring	$450^{o}F$ exceeded during run

-Continued-

TABLE 8. TROUBLESHOOTING AND REPAIR

Component	General Remarks	Problem	Repair Sequence
MRI impactor		Melted O-ring	Viton O-rings not used
			Replace with Viton O-rings
		Plugged jet plates	Use copper wire to clean
			Clean in soap and water, and rinse with DI H_2O and blot dry.
CCS filter holder	The G/R filter holder is made out of quartz and especially when it is hot, mechanical shocks will cause breakage. The filter holder is designed to always be run with a filter on the quartz frit. Because of the high temperatures employed, greasing the joints is not recommended.	No seal to filter	Check extension tube. If it is not making a seal, have the glass blower repair. As a temporary repair, a washer out of tissuequartz can be used to promote a seal.
		Gas leakage	Check thermocouple well for pinhole leak.
			Check alignment of ball and socket joints. Try to maintain linearity.
			Check seal at joints, clean joints, and retest.
			Check joints for thermal warping. Replace.
		Plugged frit	Soak in hot chromic acid cleaning bath for 12 hours. Rinse with DI H_2O till neutral.
CC coil	The coil is an especially delicate piece of equipment. Clear visibility of the coils is necessary, so maintain the water jacket's cleaniness.	Gas leakage	Check thermocouple well for pinhole leak.
			Check alignment of ball and socket joints. Try to maintain linearity.
			Check seal at joints, clean joint, and retest.
			Check joints for thermal warping. Replace.
			Soak in hot chromic acid cleaning both for 12 hours. Rinse with DI H_2O till neutral.

-Concluded-

APPENDIX A

Isokinetic Flow Rate Calculator Instructions

AEROTHERM
ACUREX Corporation

OPERATING MANUAL

ISOKINETIC FLOW RATE CALCULATOR

P10-01

AEROTHERM DIVISION
ACUREX CORPORATION

485 Clyde Avenue

Mountain View, California 94042

415-964-3200

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Consider a source test under the following parameters:

$C_p = 0.85$ pitot tube coefficient

$\Delta H@ = 1.95$ orifice calibration factor, supplied by train manufacturer or determined experimentally (page 6)

$\%H_2O = 20$ Percent water by volume present in stack gas

$T_m = 75^{\circ}F$ Temperature at the dry gas meter

$P_s/P_m = 1.1$ Ratio of STACK Pressure (Ps) to Pressure at the meter (Pm)

$T_s = 650^{\circ}F$ Temperature of stack gas

$\Delta P_{avg} = 0.2"H_2O$ Average Pitot reading taken on preliminary traverse

to be determined
{ D_n Nozzle diameter (inches)
{ ΔH Orifice pressure differential
{ ΔP Individual Pitot reading

Detailed Instructions

1. Set Cp over ΔH@

Place the cursor on the correct value of Cp (pitot tube coefficient) for your train. If Cp has not been calibrated, assume 0.85 for an S or reverse type pitot, and 0.99 for a standard type pitot tube. Almost all EPA type trains employ an S type pitot tube.

Move the top slide until the correct value of ΔH@ (orifice calibration factor) is directly under the Cp value. In our example, 1.95 would be placed under 0.85. If the train has variable orifices or very unusual calibration factor, see page 10.

2. Set % H_2O to Reference

Move the cursor to the reference arrow. Then move the second body slide so the correct %H_2O is under the reference arrow. (%H_2O is either measured directly from a previous experiment or is estimated. A variation of ± 1% is acceptable.)

In our example, 20% is placed below the arrow.

3&4 Read Index at arrow/set Tm at Index Number

To facilitate transferring numbers from one slide to another, an index (number line) has been provided. Read the index at the reference arrow, then slide Tm to that number. A faster method is to move the hairline over the reference arrow and then slide Tm to the hairline. In our example, the hairline would be moved to the reference arrow and 75°F then slid to the hairline.

5&6 Read Second Index Number at Ts/Set Ps/Pm at Second Index Number

The number line may be read again as in 3 & 4 or using the more rapid method: Move the hairline to Ts, then set Ps/Pm to the hairline. Be sure hairline does not move.

7. If Dn is not known, set the average ΔP reading under the reference arrow C on the ΔH scale. Be sure the B reference arrow does not move (hold this point with the hairline if desired). Read the exact nozzle size under the B arrow. In our example, this is 0.380 inches. We would select a 3/8" nozzle (0.375 inches) and move the Dn scale until 0.375 is directly under the reference arrow B.

8. Read ΔH setting opposite ΔP reading using cursor as needed.

The proportional ratio between ΔH and ΔP has now been set, and any value of ΔP is now directly under the correct setting of ΔH.

For our example:

ΔP	ΔH
1	8.8
.5	4.4
.2	1.76
.1	.88

The bottom slide is designed to be tightly held in the calculator body so that the ΔP/ΔH ratio does not slip during use. Should this loosen through use, a piece of tape on the back side will again tighten it.

Additional Thoughts:

Rapid Use of the Calculator

With experience, all the operation and settings can be done in less than 25 seconds. We suggest the following sequence for maximum speed.

1. Set Cp over $\Delta H@$
2. Move hairline to arrow, then set %H_2O to hairline
3. Move hairline to next arrow
4. Set Tm to hairline
5. Move hairline to Ts
6. Set Ps/Pm to hairline
7. Set hairline to B
8. Set avg. ΔP to arrow C
9. Select nozzle size and move to hairline
10. Read ΔP vs. ΔH

(all that in 25 seconds!)

Resetting calculator

At times, variables may change during the course of a test necessitating readjustment of the calculator. The following is presented as a guideline:

Cp	should not change
$\Delta H@$	should not change
%H_2O	± 1%
Tm	± 10°F
Ts	± 25°F
Ps/Pm	± 1%

Rapid temperature change: If the stack temperature (T_s) changes significantly during the course of the test (±25 - 50°F), the calculator may be reset rapidly without repeating the entire calculation as follows:

A. Place the hairline over the new T_s and move old T_s to the hairline.
B. Move the actual nozzle size to reference Arrow B if the old nozzle size will still produce reasonable flow rates. Otherwise, select a new nozzle.
C. Read ΔH across from ΔP as before.

WORKED EXAMPLE

	Example 1	2	3	4	5	6	7
Cp	.85	.85	.85	.85	.85	.99	.85
ΔH@	1.95	1.95	1.95	3	1.95	1.95	1.95
$\%H_2O$	30	0	30	30	30	30	30
Tm	100	100	100	100	0	100	100
Ts	500	500	1500	500	500	500	500
Ps/Pm	1.1	1.1	1.1	1.1	1.1	1.1	.8
Dn (exact)	.383	.330	.735	.230	.506	.355	.276
Dn (actual)	.375	.375	.750	.250	.5	.375	.250
Avg. ΔP	.2	.2	.03	1.0	.08	.2	1.0
ΔP_1	1.0	.1	.1	1.0	.1	.1	1.0
ΔH_1	8.5	1.54	6.67	2.59	2.21	1.15	1.22
ΔP_2	.1	.01	.01	.1	.01	.01	.1
ΔH_2	.85	.154	.667	.259	.221	.115	.122

A constriction in a tube in which a gas is flowing causes a difference in pressure between the upstream and downstream sides of the constriction. This pressure differential is related to the rate of flow.

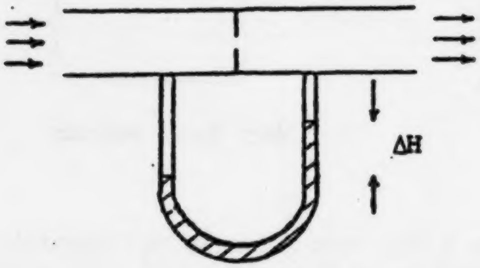


Figure I. Orifice Meter

An orifice meter, Figure I., is a type of constriction which uses the following relationship between the flow rate and the pressure differential to measure the rate of flow:

$$Q_m = K_m \sqrt{\frac{T_m \ \Delta H}{P_m \ M_m}}$$

where Q_m = volumetric gas flow rate

T_m = gas temperature, absolute

M_m = molecular weight of the gas

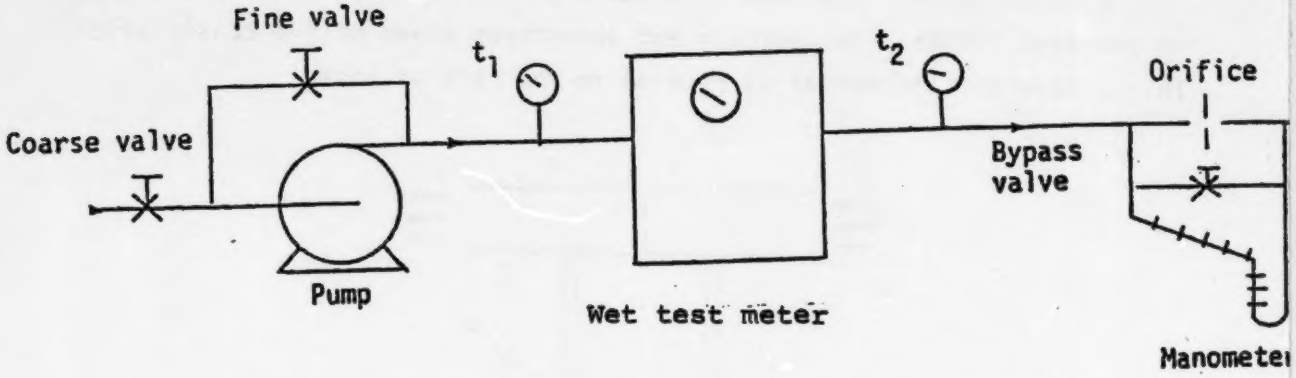
P_m = pressure of the gas, absolute

ΔH = the pressure drop across the orifice

K_m = a proportionality factor

subscript m = refers to the meter

FLOW DIAGRAM



Note that although a dry test meter is not normally used as a primary standard for calibrating a flow meter, if your train meter has been calibrated to within 1%, it may be used as a laboratory calibration.

Procedure

1. Level manometer by leveling meter box.
2. Zero orifice leg of the manometer with the manometer bypass valves "out".
3. Turn on pump.
4. Turn manometer bypass valves "in".
5. Adjust the coarse and fine valves to get a reading of 0.5 inches of water on the manometer leg (ΔH).
6. Start stopwatch at same time you read dry test meter volume (V_1). Let dry test meter rotate at least one full revolution. (The longer the time, the better your accuracy.)
7. Stop watch and read the dry test meter (V_2) simultaneously.
8. Read t_1 and t_2.
9. Record ΔH, V_1, V_2, θ, t_1, and t_2.
10. Repeat steps 6 through 9, but adjust for new ΔH of 1.0, 2.0, and 6.0 inches of water on the manometer leg. (With some units, it may be possible to reach only 3 or 4 inches of water.)

Data

Table I. Orifice Calibration Data Sheet

Meter Box No. ___								
Δm in. H_2O	V_1 cf	V_2 cf	θ min	t_1 °F	t_2 °F	$V_2 - V_1$ cf	Q_m cfm	K_m
0.5								
1.0								
2.0								
6.0								

Calculations

1. Calculate Q_m as follows:

$$Q_m = \frac{V_2 - V_1}{\theta} \left[\frac{t_2 + 460}{\frac{t_1 + t_2}{2} + 460} \right]$$

2. Calculate K_m for each Δm as follows:

$$K_m = Q_m \sqrt{\frac{P_m M_m}{T_m \Delta H}}$$

$P_m = P_{atm}$

$M_m = M_{(air)} = 29$

$T_m = t_2 + 460$

3. Calculate the average K_m as follows:

$$\overline{K_m} = \frac{\Sigma K_m}{4}$$

Using the Average Km, compute ΔH@ * from the following:

$$Qm = Km \sqrt{\frac{Tm \quad \Delta H@}{Pm \quad Mm}}$$

Where Qm = 0.75 ft.^{3}/min
Tm = 70°F = 530°R
Pm = 29.92 in Hg
Mm = 29.0 lbs/lb.mole
Km = experimentally determined

Use Qm = 0.75 ft.^{3}/min if this flow is within the measured range of the orifice. (For example, avg. experimental Qm ≈ 0.75.) This is a typical EPA type orifice.

If the average value of Qm is significantly different from 0.75 ft.^{3}/min, then assume a new standard flow rate for calibration purposes.

For example, a large orifice may have a ΔH@x of 1.5 inches of water at 4 scfm dry air.

* ΔH@ is a symbol that identifies the orifice flow characteristics. It is "defined" as the pressure drop in inches of H_2O across an orifice at standard conditions (70°F, 1 Atmos.) dry air flowing through at the rate of 0.75 cfm.

Should any other flow rate or conditions be employed, this should be clearly indicated.

ALTERNATE CALIBRATION PROCEDURES

DRY GAS METER AND ORIFICE METER

Connect the components as shown in Figure 20. The wet test meter is a 1-cubic-foot-per-revolution meter with ± 1 percent accuracy. Run the pump for about 15 minutes with the orifice manometer set at about 0.5 inch of water to allow the pump to warm up and to permit the interior surface of the wet test meter to be wetted. Then gather the information as requested on the data sheet in Figure 9. Calculate γ, the ratio of accuracy of the wet test meter to the dry test meter, and $\Delta H_@$. If an average γ of 1.0 ± 0.01 is not obtained, the dry gas meter should be

Date ________________________ Box No. ________________

Barometric pressure, P_b = ___in. Hg Dry gas meter No. ________________

Orifice manometer setting, ΔH, in. H_2O	Gas volume wet test meter V_w, ft^3	Gas volume dry gas meter V_d, ft^3	Temperature				Time θ, min		$\Delta H_@$
			Wet test	Dry gas meter					
			Meter t_w, °F	Inlet t_{di}, °F	Outlet t_{do}, °F	Average t_d, °F			
0.5	5								
1.0	5								
2.0	10								
4.0	10								
6.0	10								
8.0	10								
						Average			

Calculations

		γ	$\Delta H_@$
ΔH	$\frac{\Delta H}{13.6}$	$\frac{V_w P_b (t_d + 460)}{V_d\left(P_b + \frac{\Delta H}{13.6}\right)\left(t_w + 460\right)}$	$\frac{0.0317\ \Delta H}{P_b\ (t_d + 460)}\left[\frac{(t_w + 460)\ \theta}{V_w}\right]^2$
0.5	0.0368		
1.0	0.0737		
2.0	0.147		
4.0	0.294		
6.0	0.431		
8.0	0.588		

γ = Ratio of accuracy of wet test meter to dry test meter. Tolerance = ± 0.01

$\Delta H_@$ = Orifice pressure differential that gives 0.75 cfm of air at 70° F and 29.92 inches of mercury, in. H_2O. Tolerance - ± 0.15

Figure 9. Suggested orifice and dry gas meter calibration and calculation form.

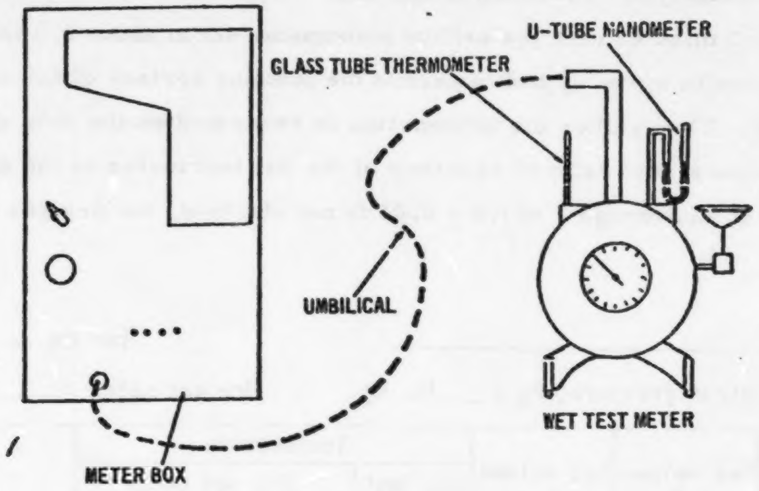

U-TUBE MANOMETER
GLASS TUBE THERMOMETER
UMBILICAL
WET TEST METER
METER BOX

Figure 20. Calibration setup.

adjusted until γ meets the specification. This can be accomplished by removing the plate on top of the gas meter and adjusting the linkages.

High Volume Samplers/Variable Orifice Calibrations

The IFRC is designed for isokinetic sampling trains based on the original EPA design specifications which limits maximum sampling rates to about1.5cfm. However, certain experimental conditions (extremely low grain loading, very irregular operation, etc) may require taking a very large sample or sampling for only a very short period of time. High volume samplers have been developed to fill this void. If the sampler has the basic EPA design but simply has larger pumps, impingers, and other components, the CSI calculator may be used directly for isokinetic calculations.

Because of the increased flow rates, these high volume trains are frequently provided with a series of orifices to monitor the flow rate out of the gas meter. The only change needed to use the CSI calculator for any orifice is to obtain a correct ΔH@ for each orifice.

1. Obtain ΔH@ for each orifice. If ΔH@ is unknown, proceed to page 6; if ΔH@ is determined at a flowrate other than 0.75scfm, proceed to #3; if ΔH@ is determined at 0.75 scfm, proceed to #2.

2. Input ΔH@ in normal fashion as described in the condensed instructions on the calculator body.

3. If ΔH@ has been obtained at any other flowrate (flow ≠0.75scfm), obtain a new ΔH@ by using the following equation.

$$\Delta H@' = \Delta H@_x \left[\frac{.75}{x}\right]^2$$

Where $H@x$ = orifice differential pressure (inches H_2O) at x scfm using dry air

Using this new ΔH@′ proceed as before with normal operation of the calculator.

If an extremely high flow rate is used and ΔH@′ does not fall on the printed scales, the calculator may still be used. For example, say ΔH@′ is found to be 0.05 which does not lie on the ΔH@ scale, but should lie somewhere to the right of ΔH@ = 0.1. Simply move the decimal point one place to the right (ΔH@' = 0.05 → 0.5) and use this ΔH@′ in the normal fashion until step 8 (sizing the nozzle). Instead of reference arrow B, there is a small tick mark approximately 2 inches to the left of reference arrow. Use this as the new reference B point and proceed as before. Using the new reference point automatically takes out the factor of 10 that was introduced when we arbitrarily shifted the decimal point in the ΔH@' , but allows the majority of the computations to be performed in the center of the calculator body.

EXAMPLES

Example 1:

$\Delta H@$ is calibrated for a large orifice as 2.0" H_2O at 3cfm dry air @ STP 70°F,

$$\Delta H@' = 2\left[\frac{.75}{3}\right]^2 = 0.125$$

Use 0.125 for $\Delta H@$ when testing with this orifice.

Example 2:

$\Delta H@$ is calibrated for a very large orifice as 1.89" H_2O at 6cfm

$$\Delta H@' = 1.89\left[\frac{.75}{6}\right]^2 = 0.029$$

Set 0.29 for $\Delta H@$ (note decimal shift) but also use the small mark to the left of arrow B as reference B.

VARIABLE MOLECULAR WEIGHTS

The CSI isokinetic flowrate calculator is designed for use in systems where M_d (the dry molecular weight of the gas) is approximately 29. Situations may arise, primarily in a process stream, where M_d will be considerably different from 29. Two corrections will be necessary to adjust this calculation for use in gases differing significantly from 29 g/mole.

The orifice calibration coefficient (ΔH@) is normally computed for a flow rate of dry air at 0.75 scfm (70^oF and 760 mm Hg). Sampling in a gas stream with $\Delta M_d \neq 29$ will, of course, change this calibration.

The simplest way to determine the new ΔH@ is to repeat the calibration as given in section "Calibrating Orifice Meters" using the actual stack gas as the source and using the correct M_d in equation on page 9. In lieu of this experiment, a new ΔH@ may be approximated from the following expression.

A. $\Delta H@\ (M_d = x) = \Delta H@\ (M_d = 29) \left(\frac{x}{29}\right)$

This new $\Delta H@\ (M_d = x)$ may be used directly in the calculator.

B. A second correction to account for the difference in M_d in the basic isokinetic equations is made through the $\%H_2O$ scale. Using Figure M_s1 find the actual $\%H_2O$ in the system, then move up to the curve representing the actual M_d. Finally, locate a new $\%H_2O\ (M_d = x)$ on the ordinate. Use this new $\%H_2O$ in the normal fashion in the calculator.

The isokinetic sampling may now proceed in the normal fashion.

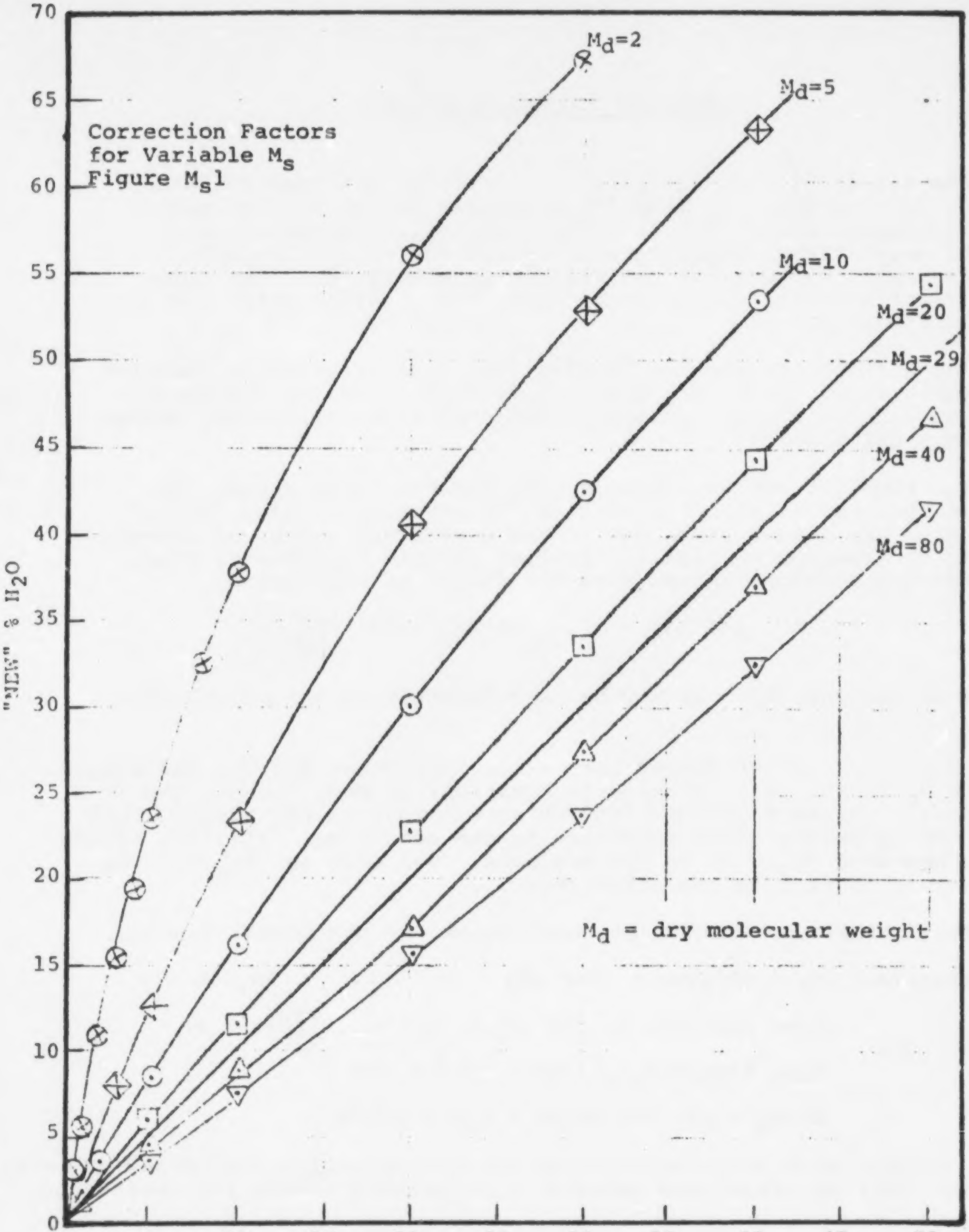
Example: $M_d = 20$ g/mole $\Delta H@\ (M_d = 29) = 2.0$ $\%H_2O = 15\%$

From equation A, $\Delta H@\ (M_d = 20) = 2.0\left(\frac{20}{29}\right) = 1\ 34$

From Figure M_s1, Actual $\%H_2O = 15\%$

At $M_d = 20$, the "New" $\%\ H_2O = 17.5\%$

Continue with computation as in previous examples. Should $\Delta H@\ (M_d = x)$ not fall on scale, see section on variable orifices for assistance.



Correction Factors for Variable M_s

Figure M_s1

APPENDIX B

Derivation of H_2SO_4 ppm Calculation Equation

Acid/Base Titration

1. Calculate the number of moles titrated

$$\text{moles} = \frac{NV \times 10^{-3}}{2} \qquad (1)$$

where N = Normality of base
V = Volume of base used (ml)

2. The number of moles in the original sample are:

$$\text{moles} = \frac{(NV \times 10^{-3})}{2}\,\frac{50}{A} = (2.5 \times 10^{-2})\,\frac{NV}{A} \qquad (2)$$

where A = aliquot taken from 50 ml sample

3. Volume of acid at 21°C(70°F) and 1 atm (29.92 in Hg)

$$PV = nRT$$

$$V_{H^+} = \frac{\left(\frac{2.5 \times 10^{-2}\ NV}{A}\right)RT}{P} \qquad (3)$$

$$V_{H^+} = \frac{6.03 \times 10^{-1}}{A}$$

where P = pressure (1 atm)
R = gas constant (0.08205 atm · liters/°K · mole)
T = temperature (293°K)

4. Volume of gas sampled at STP (liters):

$$V_G = V_S\ 28.32\ \left(\frac{530}{460+t}\right)\left(\frac{P_m}{29.92}\right) = V_S\ 501.7\ \left(\frac{P_m}{460+t}\right) \qquad (4)$$

where V_G = liters of gas at 21°C (70°F) and 1 atm (29.92)
V_S = gas meter volume (cu. ft.)

t = dry test meter temperature (°F)

P_m = meter pressure (in. Hg.)

5. ppm H_2SO_4 (Vol./Vol.):

$$\text{ppm } H_2SO_4 = \frac{V_{H^+} \times 10^{+6}}{V_G}$$

$$= (1{,}201.9)\left(\frac{NV\ (460+t)}{A\ V_S\ P_m}\right)$$

6. For the sulfate titration an additional factor of ten is added to equation 5 to correct for the extra dilution due to the ion exchange column. Also molarity of the $Ba(ClO_4)$ solution is used in place of the normality of the NaOH solution. For the sulfate titration:

$$\text{ppm } H_2SO_4 \text{ (Vol/Vol)} = 12{,}019\left(\frac{MV(460+t)}{A\ V_S\ P_m}\right) \qquad (6)$$